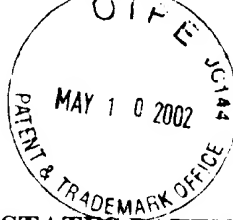


Atty. Docket: 40018160.0003



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Reissue Patent Application of)
Inventors: TOMOHIRO KAWASE et al.) Group Art: 1765
Serial No.: 09/824,965) Examiner: Robert Kunemund
Filed: April 3, 2001)
Title: Method Of Preparing Group III-V)
Compound Semiconductor Crystal)

CERTIFICATE OF MAILING UNDER 37 C.F.R. § 1.8

I hereby certify that this correspondence is being deposited with the United States Postal Service, with sufficient postage as first class mail, in an envelope addressed to Assistant Commissioner for Patents, Washington, DC 20231 and mailed on the below date of mailing.


Name: Patricia McEllen

4/30/02
Date of Signature and Mailing

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ORIGINALLY FILED

ATTENTION: Director of Technology Center 1700
Assistant Commissioner for Patents
Washington, D.C. 20231

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TC 1700

PROTEST UNDER 37 C.F.R. 1.291

Dear Sir:

This Protest under 37 C.F.R. § 1.291 is being submitted against the above-identified reissue application. 37 C.F.R. § 1.291 sets forth the following requirements of a Protest for consideration by the Examiner:

§1.291 Protests by the public against pending applications.

(a) Protests by a member of the public against pending applications will be referred to the examiner having charge of the subject matter involved. A protest specifically identifying the application to which the protest is directed will be entered in the application file if:

(1) The protest is submitted prior to the date the application was published or the mailing of a notice of allowance under §1.311, whichever occurs first; and

(2) The protest is either served upon the applicant in accordance with § 1.248, or filed with the Office in duplicate in the event service is not possible.

(b) Protests raising fraud or other inequitable conduct issues will be entered in the application file, generally without comment on those issues. Protests which do not adequately identify a pending patent application will be returned to the protestor and will not be further considered by the Office. A protest submitted in accordance with the second sentence of paragraph (a) of this section will be considered by the Office if the application is still pending when the protest and application file are brought before the examiner and it includes:

(1) A listing of the patents, publications, or other information relied upon;

(2) A concise explanation of the relevance of each listed item;

(3) A copy of each listed patent or publication or other item of information in written form or at least the pertinent portions thereof; and

(4) An English language translation of all the necessary and pertinent parts of any non-English language patent, publication, or other item of information in written form relied upon.

(c) A member of the public filing a protest in an application under paragraph (a) of this section will not receive any communications from the Office relating to the protest, other than the return of a self-addressed postcard which the member of the public may include ends with the filing of the protest, and no further submission on behalf of the protestor will be considered, except for additional prior art, or unless such submission raises new issues which could not have been earlier presented.

Timing of Protest Under 37 C.F.R. § 1.291(a)(1)

37 C.F.R. § 1.291(a)(1) states that a protest will be entered if the "protest is submitted prior to the date the application was published or the mailing of a notice of allowance under § 1.311, whichever occurs first." For reissue applications, the M.P.E.P. § 1441.01 explains that

A protest with regard to a reissue application should be filed within the 2-month period following the announcement of the filing of the reissue application in the *Official Gazette*. If the protest of a reissue application cannot be filed within the 2-month delay period, the protest can be submitted at a later time. Where the protest is submitted after the 2-month period, no petition for entry of the protest under 37 CFR 1.182 is needed with respect to the protest being submitted after the 2 months *unless a final rejection has been issued or prosecution on the merits has been closed for the reissue application*. [Italics added]

Where the protest is submitted after the 2-month period, the protest may be received after the first Office action by the examiner, since reissue applications

are taken up 'special.' Once the first Office action is mailed (after the 2-month period), a member of the public may still submit pertinent information in the form of a protest under 37 CFR 1.291(a), and the examiner will consider the information submitted in the next Office action, to the extent that such consideration is appropriate.

The above-referenced reissue application was published in the *Official Gazette* of July 17, 2001. However, under M.P.E.P 1441.01, a Protest is timely in a reissue application if filed before "a final rejection has been issued or the prosecution on the merits has been closed." Neither has occurred in this reissue application, and, in fact, a first (non-final) Office Action was mailed on March 6, 2002. A Response to that Office Action was entered recently entered on April 15, 2002. The Protest is therefore timely under § 1.291(a)(1).

Service of Protest Under 37 C.F.R. § 1.291(a)(2)

37 C.F.R. § 1.291(a)(2) requires that the Protest be "either served upon the applicant in accordance with § 1.248, or filed with the Office in duplicate in the event service is not possible." A Proof of Service is being filed concurrently herewith showing service of this Protest upon the Applicant in accordance with § 1.248. A copy of that Proof of Service is attached as Exhibit 1 to this Protest.

Requirements of Protest Under 37 C.F.R. § 1.291(b)

37 C.F.R. § 1.291(b) requires a protest to contain:

- (1) A listing of the patents, publications, or other information relied upon;
- (2) A concise explanation of the relevance of each listed item;
- (3) A copy of each listed patent or publication or other item of information in written form or at least the pertinent portions thereof; and
- (4) An English language translation of all the necessary and pertinent parts of any non-English language patent, publication, or other item of information in written form relied upon.

(1) Listing Of Patents, Publications Or Other Information Relied Upon

A PTO Form 1449 is attached to this Protest as Exhibit 2 listing the patents, publications and other information relied upon. There is one item listed in the PTO Form 1449 and is reproduced here for convenience:

1. European Patent Application with Publication No. 0 417 843 A2 published March 20, 1991 to inventor Russell Eugene Kremer ("Kremer EP Reference") attached as Exhibit 3.

(2) A Concise Explanation Of The Relevance Of Each Listed Item

A. Kremer EP Reference

The Kremer EP Reference discloses a vertical Bridgman ("VB") process designed to alleviate two problems of the past: (1) that the monocrystalline ingot has a tendency to adhere to the crucible surface (page 4, lines 23-41) and (2) that controlled carbon doping in a VB process is difficult since carbon sources are isolated from the monocrystalline compounds grown in the sealed ampoule.

The solution disclosed by the Kremer EP Reference is to use a powdered solid (preferably boron nitride) in the crucible to solve the adhesion problem (page 6, lines 26-33) due to the higher melting point of the boron nitride. With regard to the carbon doping problem, the Kremer EP Reference teaches placing a carbon source outside of the crucible and in "fluid communication" with the material inside the crucible (page 6, lines 11-25).

The invention described by the Kremer EP Reference is performed using the VB method ("[t]he present invention will be described mainly in terms of the production of monocrystalline gallium arsenide (GaAs) by a vertical Bridgman process[;]" page 5, lines 38-42)). The Kremer EP Reference explains the basic principles of the VB method in the Background of the Invention as well as in the Detailed Description. In particular, the Kremer EP Reference is clear that the

VB method has tried to use B_2O_3 (boric oxide) to solve the problem of crystal adhesion (page 4, lines 30-37) with limited success. The Kremer EP Reference then goes on to provide three Examples of the process of the invention.

Example 1 uses the VB method with the boron nitride coating on the crucible to solve the adhesion problems of the prior art. The example produced ingots that "were easily removed from the crucible" (page 7, line 15) and summarized other electrical characteristics in Table 1 when compared to other methods of manufacturing substrates.

Example 2 then focused on the carbon-doping of the GaAs. Here, Example 2 first compares the well-known prior art method of "LEC" that exposed the GaAs to graphite and controls the amount of carbon doping using B_2O_3 (boric oxide) (page 7, lines 53-56). In this Example 2, the VB method was used again except a thick carbon disk was placed on top of the crucible. (Page 8, line 4).

Example 3 then uses the VB method of Example 2 once again for controlling the carbon-doping process to provide ingots A-H, but then uses a different process for another ten ingots, ingots I-R. Specifically, the ten ingots had graphite powder added directly into the crucible (page 8, line 26). The Kremer EP Reference then compared the VB method used for Examples 1, 2 and the eight ingots of Example 3 to the ten ingots of Example 3 to conclude that the present invention method provides "superior uniformity of carbon doping." (Page 8, lines 28-30).

(1) Invalidity of Claim 1 under 35 U.S.C. 102 (b)

35 U.S.C. 102(b) states:

35 U.S.C. 102. Conditions for patentability; novelty and loss of right to patent.
A person shall be entitled to a patent unless

...

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States, or

To show that claim 1 is invalid under 102(b), each element of claim 1 must be expressly or inherently found in a single prior art reference as explained by M.P.E.P. § 2131:

TO ANTICIPATE A CLAIM, THE REFERENCE MUST TEACH EVERY ELEMENT OF THE CLAIM

'A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference.' *Verdegaal Bros. v. Union Oil Co. of California*, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987). 'The identical invention must be shown in as complete detail as is contained in the ... claim.' *Richardson v. Suzuki Motor Co.*, 868 F.2d 1226, 1236, 9 USPQ2d 1913, 1920 (Fed. Cir. 1989). The elements must be arranged as required by the claim, but this is not an *ipsissimis verbis* test, i.e., identity of terminology is not required. *In re Bond*, 910 F.2d 831, 15 USPQ2d 1566 (Fed. Cir. 1990). Note that, in some circumstances, it is permissible to use multiple references in a 35 U.S.C. 102 rejection. See MPEP § 2131.01.

M.P.E.P. § 2131. Claim 1 of the reissue application reads:

1. A method of preparing a carbon-doped group III-V compound semiconductor crystal, comprising the steps of:

placing a compound raw material, solid carbon, and a boron oxide substance into a crucible or a boat,

sealing said crucible or boat containing said compound raw material, said solid carbon, and said boron oxide substance within an airtight vessel formed of a gas impermeable material,

heating and melting said compound raw material in said crucible or said boat sealed within said airtight vessel, and

solidifying said melted compound raw material to grow a carbon-doped compound semiconductor crystal,

wherein an amount of said solid carbon placed into said crucible or said boat is larger than an amount of carbon doped into said compound semiconductor crystal.

Each of the elements of claim 1 is either expressly or inherently described in the Kremer EP Reference as follows.

(a) **A method of preparing a carbon-doped group III-V compound semiconductor crystal, comprising the steps of:**

Example 2 and Example 3 clearly describe a “process of the instant invention” for “carbon doping” of GaAs, a Group III-V compound semiconductor compound. (Page 7, lines 50-57 and Page 8, lines 23-30). In fact, the title of the invention for the Kremer EP Reference is “Process for producing monocrystalline group II-V or group III-V compounds and products thereof.” (Page 1, Title). This limitation is therefore expressly described in the Kremer EP Reference.

(b) **placing a compound raw material, solid carbon, and a boron oxide substance into a crucible or a boat,**

A “compound raw material” is described in the Kremer EP Reference as a “polycrystalline material” and is “placed in the remainder of the crucible.” (Page 6, lines 33-34). This step is further explained as step (b) on page 5 of “placing an amount of said compound in its polycrystalline form into said coated crucible.” (Page 5, line 3). This element is therefore expressly described in the Kremer EP Reference.

“Solid carbon” (in the form of graphite powder) is placed “directly in the crucible” in Example 3. (Page 8, line 26). While Example 3 compares the process of the invention (carbon doping with carbon located outside of the crucible) of Examples 1 and 2 to the method of Example 3 (carbon doping with solid carbon directly in the crucible) (page 8, lines 28-30), Example 3 nevertheless discloses that solid carbon may be placed directly in the crucible as recited in claim 1. Merely because a reference disparages an element does not render the reference less anticipatory. (“A reference is no less anticipatory if, after disclosing the invention, the reference then disparages it. Thus, the question whether a reference ‘teaches away’ from the invention is inapplicable to an

anticipation analysis.” *Celeritas Technologies, Ltd. v. Rockwell International Corporation*, 150 F.3d 1354, 1361 (Fed. Cir. 1998), citing *Kalman v. Kimberly-Clark Corp.*, 713 F.2d 760, 781 (Fed. Cir. 1983)). This element is therefore expressly described in the Kremer EP Reference.

“Boron oxide” is clearly described in the Kremer EP Reference with regard to the VB method and the Liquid Encapsulated Czochralski (LEC) method. (“[A reference] suggests the use of boric oxide B_2O_3 to suppress decomposition and evaporation of arsenic from molten and crystalline GaAs during vertical Bridgman processes” (page 4, lines 31-34); “The amount of carbon incorporated into LEC-produced GaAs may be controlled by varying the water content of the B_2O_3 encapsulant used in the process (page 7, lines 54-55)). As argued by the Applicants in an information disclosure statement, the remaining issue is whether the combination of boron oxide and solid carbon in the crucible is described in the Kremer EP Reference. (The Applicants argue that, “[w] hile the prior art discussion in the Kremer US patent and Kremer EP application do refer to B_2O_3 and carbon powder placed in the crucible, there is no statement in either reference disclosing, teaching or suggesting a combination including both B_2O_3 and carbon powder placed in the crucible.” (Second IDS, Exhibit 4, page 4)

First, M.P.E.P. § 2131 clearly has no “combination” requirement, finding a reference anticipatory if “each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference.” This legal requirement is clearly met since both elements are described in the single Kremer EP Reference. Second, there is a suggestion to combine carbon powder and B_2O_3 in the Kremer EP Reference: the Kremer EP Reference uses the same VB method (page 5, lines 38-39) in Example 3 (teaching solid carbon (page 8, line 26)) and in the Background of the

Invention (teaching boric oxide (page 4, line 33)). That same VB method provides the suggestion to combine these two elements to one skilled in the art and thus these limitations of claim 1 are met.

Applicants further argue in the Second IDS (Exhibit 4, page 3) that it is improper "to combine two different parts of a publication to form an anticipatory reference unless the two parts are related" citing *Ecolochem, Inc. v. Southern Cal. Edison Co.*, 227 F.3d 1361, 1368-69 (Fed. Cir. 2000). In response, it is noted that both B₂O₃ and solid carbon are "related" in the Kremer EP Reference since both are expressly used in the same VB method. A person skilled in the art of the VB method would consider combining these two elements from the Kremer EP Reference since both of these elements are described for use in the same VB method. Further, *Ecolochem* (attached as Exhibit 5) has no holding that elements must be "related" to be anticipatory as asserted by Applicants. Rather, that case purely turned on factual issues specific to that case. The Federal Circuit made no holding in that case that, as a matter of law, two elements in a 102 (b) reference must be "related" to be anticipatory.

- (c) sealing said crucible or boat containing said compound raw material, said solid carbon, and said boron oxide substance within an airtight vessel formed of a gas impermeable material,

This element is described in the Kremer EP Reference by the sealing of the crucible-ampoule assembly containing all the materials claimed. (Page 6, lines 35-37).

- (d) heating and melting said compound raw material in said crucible or said boat sealed within said airtight vessel, and

This element is described in the Kremer EP Reference by employing heating elements of a furnace to reduce the material to a melt. (Page 6, lines 38-41).

(e) solidifying said melted compound raw material to grow a carbon-doped compound semiconductor crystal.

This element is described in the Kremer EP Reference by moving "furnace 17 slowly [] up...parallel to the vertical axis of crucible 12 to produce a solid monocrystalline ingot." (Page 6, lines 41-46).

(f) wherein an amount of said solid carbon placed into said crucible or said boat is larger than an amount of carbon doped into said compound semiconductor crystal.

The Kremer EP Reference provides the "amount of carbon doped into said compound semiconductor crystal" in Table II for ingots I-R that were manufactured using the process of Example 3 (with graphite powder added directly into the crucible). Those doping concentrations are reproduced here:

Ingot	Front (x 10 ¹⁵ cm ⁻³)	Tail (x 10 ¹⁵ cm ⁻³)
I	0.5	<0.1
J	1.5	<0.1
K	0.1	<0.1
L	4.0	0.6
M	0.9	0.5
N	1.1	<0.1
O	1.2	0.2
P	1.2	0.5
Q	0.7	<0.1
R	0.4	<0.1

Page 9, Table II.

To determine the "amount of said solid carbon placed into said crucible" for ingots I-R of Example 3, the language of Example 3 is reviewed. Example 3 states that

"the method used to produce ingots A-H was employed with the following modifications." (Page 8, lines 24-25). One modification was to use "graphite powder (carbon)" directly in the crucible. (Page 8, line 26). The other was that there was no opening in the cap since the carbon was within the crucible. (Page 8, line 27). Example 3 does not provide the specific numerical amount of graphite powder placed into the crucible.

However, Example 3 does explain that the methodology used "to produce ingots A-H" was used in Example 3 with modifications, and, in turn, that the methodology to produce ingots A-H used "the method described in Example 2" with certain modifications. (Page 8, lines 15-25). Example 2 used the method of Example 1. (Page 8, line 2). Example 1 uses a "refined version of the apparatus illustrated in FIGs. 1 and 2." From the explanation related to FIG. 2, the Kremer EP Reference explains:

...ingots of other sizes and other doping levels may be produced by this technique by varying opening 24 and/or the amount of carbon placed outside the crucible 12. For example, a gallium arsenide ingot of about 100mm diameter, and produced by the technique within the scope of this invention, may require a larger opening 24 and more carbon than that within the ranges disclosed above

(Page 6, lines 20-25)(Emphasis added). The scope of the invention described in the Kremer EP Reference therefore describes that the amount of carbon may be varied as part of the invention to obtain other carbon doping levels in the ingots. Since Example 3 expressly used the method described above with certain modifications, one of ordinary skill in the art placing the graphite powder in the crucible in accordance with Example 3 would have known from the teachings of page 6, lines 20-25 above that the amount of carbon may be varied to raise or lower the doped carbon in the ingot.

In addition, the ingots A-H of Example 3 used a 38mm diameter by 2.5 mm thick carbon disk. (Page 8, line 21). This carbon amount in the disk is larger than the resulting doping levels shown in Table II above for ingots A-H. Accordingly, one skilled in the art placing the graphite powder in the crucible in accordance with Example 3 would have known from the methodology of ingots A-H that the amount of graphite carbon placed in the crucible should be larger than the amount of carbon desired to be doped in the ingot from ingots A-H. Thus, this element of claim 1 is anticipated by the Kremer EP Reference.

(2) **Invalidity of Claim 1 under 35 U.S.C. 103(a) Based On The Kremer EP Reference**

35 U.S.C. §103 provides that:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in §102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains.

There are four factors which must be addressed in consideration and determination of obviousness under 35 U.S.C. §103:

- (A) Determine the scope and contents of the prior art;
- (B) Ascertain the differences between the prior art and the claims;
- (C) Resolve the level of ordinary skill in the pertinent art; and
- (D) Evaluate evidence of secondary considerations.¹

¹ *Graham v. John Deere*, 383 U.S. 1, 148 USPQ 459 (1966)

The scope and content of the Kremer EP Reference is set forth above with regard to the 102(b) analysis.

The only difference between the Kremer EP Reference and claim 1 is that the specific numerical amount of graphite powder placed directly in the crucible in Example 3 was not provided. However, as explained above with regard to the element of claim 1 of "wherein an amount of said solid carbon placed into said crucible or said boat is larger than an amount of carbon doped into said compound semiconductor crystal", one skilled in the art placing the graphite powder in the crucible in accordance with Example 3 would have known from the methodology of ingots A-H that the amount of graphite carbon placed in the crucible should be larger than the amount of carbon desired to be doped in the ingot from ingots A-H.

For these reasons, claim 1 is also invalid under 35 U.S.C. § 103(a).

(3) A Copy Of Each Listed Patent Or Publication Or Other Item Of Information In Written Form Or At Least The Pertinent Portions Thereof.

A copy of the Kremer EP Reference is attached to this Protest as Exhibit 3.

(4) An English Translation Of All The Necessary And Pertinent Parts Of Any Non-English Language Patent, Publication, Or Other Item Of Information In Written Form Relied Upon.

The Kremer EP Reference is in English and no translation is therefore believed to be necessary.

Conclusion

For the foregoing reasons, claim 1 of the reissue application is invalid and should be rejected.

Respectfully submitted,

SONNENSCHN NATH & ROSENTHAL

Date: April 30, 2002

By: 

Michael A. Molano
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EXHIBIT 1: PROOF OF SERVICE

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Reissue Patent Application of)	
)	Group Art: 1765
Inventors: TOMOHIRO KAWASE et al.)	
)	Examiner: Robert Kunemund
Serial No.: 09/824,965)	
)	
Filed: April 3, 2001)	
)	
Title: Method Of Preparing Group III-V Compou)	
Semiconductor Crystal)	

ATTENTION: Director of Technology Center 1700
Assistant Commissioner for Patents
Washington, D.C. 20231

PROOF OF SERVICE BY FIRST CLASS MAIL UNDER 37 CFR 1.248


Sir or Madam:

The undersigned, Michael A. Molano, declares and states:

On the 30th day of April, 2002, I served the **Protest Under 37 CFR 1.291** on attorney for Applicants in the above-referenced reissue application, by placing a true copy thereof in a sealed envelope with First Class postage thereon, fully prepaid and in accordance with 37 CFR 1.248, in the United States mail at San Francisco, California, addressed to:

John B. Pegram, Esq.
Fish & Richardson P.C.
45 Rockefeller Plaza, Suite 2800
New York, New York 10111

Executed this 30th day of April, 2002 in San Francisco, California.



Michael A. Molano
Reg. No. 39,777

EXHIBIT 2: PTO FORM 1449

EXHIBIT 3: KREMER EP REFERENCE



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) Publication number: 0 417 843 A2

(12)

EUROPEAN PATENT APPLICATION

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(51) Int. Cl.⁵: C30B 11/00, C30B 29/40,
C30B 29/48

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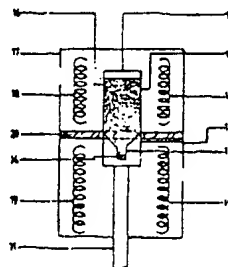
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(24) Process for producing monocrystalline group II-VI or group III-V compounds and products thereof.

(25) This disclosure relates to a process for producing monocrystalline Group II-VI or Group III-V compounds from the polycrystalline form of said Group II-VI or Group III-V compound, said process comprising coating the interior surface of a crucible with a powdered solid having a melting point higher than the polycrystalline form of the compound, placing an amount of polycrystalline compound into the coated crucible, heating the crucible to produce a melt while maintaining the powder in solid form and cooling the crucible to produce a solid compound. The preferred powdered solid is pyrolytic boron nitride. The process may be used to produce, inter alia, semi-insulating gallium arsenide having an EL2 concentration between about $0.85 \times 10^{16} \text{ cm}^{-3}$ and about $2.0 \times 10^{16} \text{ cm}^{-3}$ and a dislocation density between about 500 cm^{-2} and about 7800 cm^{-2} .

fig.1



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EP 0 417 843 A2

PROCESS FOR PRODUCING MONOCRYSTALLINE GROUP II-VI OR GROUP III-V COMPOUNDS AND PRODUCTS THEREOF

Background of the Invention

The invention relates to a process for producing monocrystalline Group II-VI or Group III-V compounds from their polycrystalline precursors.

5 Monocrystalline compounds formed by the combination of an element from Group II or III of the Periodic Table and an element from Group VI or V of the Periodic Table are critical to the electronics industry. Examples of such monocrystalline compounds are gallium arsenide (GaAs), gallium phosphide (GaP), indium phosphide (InP) and cadmium telluride (CdTe).

Monocrystalline Group II-VI compounds and Group III-V compounds may be referred to as
10 "semiconductors" ("SC") with resistivities typically within the broad range of about 10^{-3} ohm-cm to about 10^{11} ohm-cm. The conductivity of semiconductors can be varied by adding certain impurities ("dopants") to the monocrystalline material. Depending on the impurity or dopant added, semiconductors can be either n-type (excess electrons) or p-type (lack of electrons). When the Group III-V compound is GaAs, silicon is the most commonly employed n-type dopant, however, tellurium or sulfur are also useful for such
15 applications. GaAs that is heavily doped with silicon can exhibit an n-type resistivity approaching 10^{-3} ohm-cm. The most commonly employed p-type dopant is zinc, however, beryllium or carbon may also be used. For example, gallium arsenide which has been doped with carbon is mentioned in Semiconductors and Semimetals, Willardson, R.K. and Beer, A.G., Academic Press, Inc., 1984, at pages 36 and 37. However that reference does not teach methods to provide carbon doping. Zinc-doped material can also have a p-type
20 resistivity approaching 10^{-3} ohm-cm. Group III-V and II-VI semiconductors are used primarily for optoelectronic purposes such as light emitting diodes (LED's), laser diodes, photocathodes and the like and electronic applications such as transistors.

Group III-V or II-VI monocrystalline compounds which have a resistivity greater than about 10^7 ohm-cm are sometimes referred to as "semi-insulating" ("SI") semiconductors. SI semiconductors are sometimes
25 referred to as "semi-insulators". Depending on the Group III-V or II-VI compound, the monocrystalline form may be "semi-insulating" in its "undoped" or intrinsic state (e.g., GaAs, CdTe) or in a "doped" state (e.g., GaAs with chromium as a dopant or InP with iron as a dopant).

Semi-insulating GaAs can have a resistivity more than eleven orders of magnitude greater (e.g., 10^8) than that of doped semiconducting material. While the electrical properties of semiconducting material are
30 controlled primarily by the dopant(s), semi-insulating GaAs relies on the relationships among various electron energy levels. These relationships can be discussed most simply in the terms of a three-level model. The three levels are the shallow donor (or silicon) level, the shallow acceptor (or carbon) level and EL2 (or deep donor) level. The name "EL2" is associated with GaAs only and was coined by LEP (Laboratoire Electronique Philips). "E" indicates an electron trap, "L" is from LEP and "2" means that it was the second level found by LEP. In semi-insulating GaAs, EL2 dominates the charge balance, placing the
35 Fermi level near the EL2 energy level. Since the EL2 energy level is deep in the band gap, SI GaAs obtains high resistivity.

For gallium arsenide, EL2 is a defect associated with an As-on-Ga antisite and is, therefore, related to the stoichiometry of the material. If there is a very large concentration of these defects, meaning that EL2 is
40 high, fairly large concentrations of impurities can be tolerated without appreciable loss of resistivity. However, when there are relatively few defects associated with an As-on-Ga antisite, the material is much more sensitive to impurity concentrations. Non-uniform impurity concentrations may then lead to regions of high and low resistivity across a GaAs wafer. Moreover, the concentration of EL2 is also strongly dependent on the thermal history of the material. High temperature anneals followed by cooling can result in the
45 creation or destruction of EL2. A current, persistent problem is that present after-production modification processes (e.g., annealing) tend to be at high temperatures which are likely to significantly reduce the EL2 level and thus seriously reduce the effectiveness of the SI GaAs. Efforts to increase EL2 levels have had a negative effect on other properties of the SI GaAs, especially its structural integrity. One measure of structural integrity is "dislocation density". Dislocations can be revealed by etching the monocrystalline
50 material (typically with KOH for GaAs). Where a dislocation intersects the surface, an etch pit results. Thus, the etch pit density ("EPD") provides an indication of the dislocation density of the material. The terms "EPD" and "dislocation density" are frequently used interchangeably. For electronic and optoelectronic applications a relatively high EL2 and a relatively low EPD are desired. Prior art methods have been unable to achieve this combination of features, particularly when large diameter (e.g., greater than about 2.5 inches)

wafers are needed. Although the EL2 level dominates the electrical properties of Si GaAs, the resistivity and thus the semi-insulating character of monocrystalline GaAs can be increased by the controlled addition of dopants such as carbon. Semi-insulating GaAs is typically used for integrated circuit applications.

Various processes are known for preparing monocrystalline semiconducting compounds and monocrystalline semi-insulating semiconducting compounds from polycrystalline materials.

One method for producing a monocrystalline compound is the horizontal Bridgman (HB) process. Here the polycrystalline compound is placed in an half-cylinder-shaped "boat" having a monocrystalline seed at one end. As disclosed in Japanese Patent Document 58008-690-A, the boat may be comprised of an inner boat and an outer boat. In that reference, the inner boat is made of pyrolytic boron nitride and the outer boat is made of heat-insulating rigid material (e.g. quartz). Further, U.S. Patent 3 245 874 teaches reacting boron nitride with aluminum to form a solid lining of the aluminum-boron nitride reaction product in crucibles for use in the vacuum evaporation of aluminum.

In the HB process, the boat containing polycrystalline compound and monocrystalline seed is sealed within a quartz ampoule. Also within the ampoule, but outside the boat, is a separate amount of the more volatile component (such as arsenic) of the polycrystalline melt. The separate volatile component is present to help control the stoichiometric ratios in the final monocrystalline product. The quartz ampoule is sealed to prevent escape of the volatile compound. The polycrystalline material is heated to its molten form. A temperature gradient is then passed over the melt by physically moving a furnace horizontally from the seed to the other end of the boat to directionally solidify the material and form a monocrystalline compound. The temperature gradients in the HB process are quite low (about 1 to about 15° C/cm). Since dislocations are created by large temperature gradients, the small gradients employed in the HB process result in a monocrystalline product which has a low dislocation density (e.g., about 100 to 10000 cm⁻²). However, a problem is presented by the geometry of the HB boat. The "half-cylinder" shape of the HB boat produces a half-cylinder monocrystalline ingot. Most Si and many SC products are used in the form of circular wafers. Cutting circular wafers from half cylinder ingots is an inefficient use of material and time. Further, for consistent electrical and structural properties in each wafer, it is preferable to cut the wafers perpendicular to the growth axis. This is not practical with the "half-cylinder" shape of the HB produced ingot if circular wafers are desired.

For these and other reasons, vertical growth processes for producing monocrystalline electronic materials are being investigated.

The liquid encapsulated Czochralski (LEC) method generally comprises dipping a monocrystalline seed into molten polycrystalline material and slowly withdrawing the seed vertically through a liquid layer of some encapsulant. The melt and seed are contained inside a steel chamber at high pressure to prevent the volatile component (either Group V or Group VI) of the polycrystalline compound from leaving the melt. The temperature gradients used in the LEC process are usually quite high, on the order of 50° C/cm. or more. Consequently, LEC grown material has a large number of dislocations, e.g., 50000 to 100000 cm⁻². The LEC method shows particular utility in the production of semi-insulating material, but can also be used to produce semi conducting material if the desired impurities are added to the melt. However, the LEC method has several drawbacks. Materials produced by the LEC method tend to have very high dislocation densities and low uniformity. Diameter control is difficult. Also, capital and labor costs are high.

Another vertical growth process, the vertical gradient freeze method (VGF), places a polycrystalline material inside a vertically-oriented crucible or boat. The bottom of the crucible contains a monocrystalline seed of the Group II-VI or Group III-V compound to be produced. Generally, the polycrystalline material in the crucible is melted, then the temperature of the entire molten compound is reduced while distinct temperature gradients are maintained over various segments of the crucible. U.S. Patent 4 521 272 discloses a gradient freeze method for growing single crystal semiconductor compounds. The crucible has three distinct regions, each region having its own distinct temperature gradient. Single crystal semiconductors are formed by slowly cooling the molten material while maintaining these temperature gradients. U.S. Patent 4 404 172 discloses various apparatuses for growing single crystal semiconductor compounds by the VGF method. One group of apparatuses is comprised of a chamber which contains and volatilizes the volatile component of the compound to be grown, the chamber being in communication with the growth crucible. Other apparatuses disclosed are comprised of a crucible support means having a configuration to reduce radial heat flow and enhance axial heat flow. The importance of axial heat flow is discussed in "Crystal Growth: A Tutorial Approach" (Proceedings of the Third International Summer School on Crystal Growth, Edited by Bardsley et al., 1977, pp. 157-8 and 166-7).

An Improved Heat Exchanger Method (HEM) is disclosed in U.S. Patent 4 840 699 (Khattak et al.). According to U.S. Patent 4 840 699, the HEM employs a cylindrical heating chamber about a sealed quartz crucible containing gallium arsenide, and a heat exchanger to cool the bottom of the crucible. In the HEM,

polycrystalline gallium arsenide is melted, then the gallium arsenide melt is directionally solidified by withdrawing heat from the center bottom of the crucible via the heat exchanger while simultaneously supplying heat to the crucible walls via the heating chamber. No encapsulant is placed in the crucible. U.S. Patent 4 840 689 reports crack-free semi-insulating gallium arsenide having an EPD of 10000 to 20000 cm^{-2} and an EL2 concentration of $(4 \text{ to } 6) \times 10^{16} \text{ cm}^{-3}$.

The vertical Bridgman (VB) method is another process for preparing monocrystalline compounds of the types previously described. Generally, a VB process employs a vertical furnace having at least one hot zone and at least one cold zone. These zones are designed to provide a furnace temperature profile comprising a relatively flat hot zone and a relatively flat cold zone separated by a transition zone having a temperature gradient of about $5\text{--}20^\circ\text{C/cm}$. A vertically oriented crucible (usually constructed of pBN) adapted to contain Group II-VI or Group III-V compound is positioned within a sealed ampoule. Monocrystalline growth proceeds by slowly raising the furnace while holding the crucible-and-ampoule assembly stationary. (It is also possible to hold the furnace stationary and move the crucible-and-ampoule assembly. This, however, may cause vibration problems with the crucible-and-ampoule assembly.) In operation, a VB growth process involves (1) placing a mono-crystalline seed in the bottom of the crucible (which may have a specially adapted "seed well" for holding the seed), (2) loading polycrystalline material in the crucible, (3) placing the crucible in the ampoule, sealing the ampoule and placing this assembly on a pedestal (which may optionally rotate) inside the vertically oriented furnace described above, (4) heating the polycrystalline material, and the top portion of the monocrystalline seed, above its melting point and (5) moving the furnace up the length of the molten polycrystalline material to form a solid monocrystalline material. The monocrystalline Group II-VI or Group III-V ingot is then removed from the crucible and "siliced" into wafers for various electronic and/or optoelectronic uses.

As pointed out in the "Handbook on Semiconductor Materials, Properties and Preparation", (Series Ed. T. S. Moss, Vol. Ed. S. P. Keller, Vol. III, pp. 258-59, 2nd Ed., 1983), a continuing problem with monocrystalline growth processes which employ a crucible (sometimes referred to as a "boat") for holding the Group II-VI or Group III-V compound is that the monocrystalline ingot has a tendency to adhere to the crucible surfaces. This raises the obvious problem of removal of the ingot from the crucible. Additionally, the sites where the ingot adhered to the crucible surfaces tend to produce undesirable structural and electronic aberrations in the ingot.

"Crystal Growth: A Tutorial Approach" (cited above) at page 105 discloses the use of "soft-moulds" (especially bismuth oxide) to solve the problem of crystal adhesion. "Liquid Encapsulated, Vertical Bridgman Growth of Large Diameter, Low Dislocation Density, Semi-Insulating GaAs" (Journal of Crystal Growth, Hoshikawa et al., Vol. 94 (1989)) suggests the use of boric oxide, B_2O_3 , to suppress decomposition and evaporation of arsenic from molten and crystalline GaAs during vertical Bridgman processes. However, such processes are not entirely successful in that (1) it may still be difficult to remove the ingot from the boat, (2) the oxide has a tendency to contaminate the melt and/or (3) the oxide may stress or fracture the resulting solid ingot due to differences in the coefficient of thermal expansion.

We have found a method to overcome the above-described adherence problem in monocrystalline growth processes employing a crucible. The method is achieved without adversely affecting the electrical or structural properties of the resulting monocrystalline ingot. Additionally, we have found that the process of the current invention can be used to produce an improved semi-insulating material, particularly GaAs.

As discussed earlier, the resistivity, and thus the semi-insulating character, of monocrystalline GaAs can be increased by the controlled addition of dopants, such as carbon. In the LEC method of producing monocrystalline GaAs the exposure of the GaAs melt to graphite parts of the furnace virtually assures the presence of the carbon in the monocrystalline GaAs. The amount of carbon incorporated into the LEC-produced GaAs may be controlled by varying the water content of the B_2O_3 encapsulant typically used in the LEC process.

However, the monocrystalline compounds produced by various Bridgman processes are frequently grown in sealed ampoules and are thus isolated from graphite sources. Further, controlled carbon doping is a potential problem for all methods of producing monocrystalline compounds. Consequently, the current invention also comprises a method for controlled carbon doping of monocrystalline Group II-VI or Group III-V compounds.

55 Summary of the invention

The current invention is a process for producing monocrystalline Group II-VI or Group III-V compound from the polycrystalline form of said Group II-VI or Group III-V compound, said process comprising:

- (a) coating the interior surfaces of a crucible with a powdered solid, said powdered solid having a melting point higher than the melting point of said polycrystalline form of said compound,
- (b) placing an amount of said compound in its polycrystalline form into said coated crucible,
- (c) placing said coated crucible containing said compound into a heating means,
- 5 (d) heating said coated crucible containing said compound to produce a melt of said compound within said crucible while maintaining said powdered solid in solid powdered form, and
- (e) cooling said crucible and said compound to produce a monocrystalline compound.

The preferred powdered solid is boron nitride.

- The invention is also directed to a semi-insulating semiconducting material comprised of a monocrystal-
 10 line gallium arsenide having a neutral EL2 concentration between about $0.85 \times 10^{16} \text{ cm}^{-3}$ and about $2.0 \times 10^{16} \text{ cm}^{-3}$ and a dislocation density (or EPD) between about 500 cm^{-2} and about 7800 cm^{-2} . The neutral EL2 concentration may also be between about $0.85 \times 10^{16} \text{ cm}^{-3}$ and about $1.5 \times 10^{16} \text{ cm}^{-3}$ or about $0.85 \times 10^{16} \text{ cm}^{-3}$ and about $1.2 \times 10^{16} \text{ cm}^{-3}$.

- Further, the invention comprises a process for producing carbon-doped monocrystalline Group II-VI or
 15 Group III-V compound from the polycrystalline form of said Group II-VI or Group III-V compound, said process comprising:

- (a) placing a monocrystalline seed into a crucible,
- (b) placing an amount of polycrystalline compound into the crucible, said polycrystalline compound
 selected from the group consisting of Group II-VI compounds and Group III-V compounds,
- 20 (c) placing said crucible containing polycrystalline compound into a quartz ampoule,
- (d) placing a carbon source inside said ampoule yet outside said crucible, said carbon source being in fluid communication with said polycrystalline compound,
- (e) sealing said ampoule,
- (f) placing said sealed ampoule into a furnace,
- 25 (g) increasing the temperature of said furnace to produce a melt of said polycrystalline compound, and
- (h) decreasing the temperature of said furnace to cool said melt and produce a carbon-doped monocrystalline compound.

30 Brief Description of the Drawings

FIG. 1 is a schematic illustration of a portion of a vertical Bridgman apparatus useful in the practice of the current invention.

FIG. 2 is a detailed schematic illustration of a crucible useful in the practice of the current invention.

35 Detailed Description of the Invention

The present invention will be described mainly in terms of the production of monocrystalline gallium arsenide (GaAs) by a vertical Bridgman process. However, it is understood that such description is merely
 40 exemplary and the inventive concepts are applicable to the production of other Group III-V compounds or Group II-VI compounds by various processes which employ a crucible for containing the Group II-V or Group II-VI compound.

An apparatus useful for practicing the current invention is now described with reference to FIG. 1. The apparatus comprises ampoule 10 supported by support means 13. Support means 13 is mounted on
 45 pedestal shaft 11. In the preferred embodiment support means 13 is capable of rotating about its vertical axis. Positioned inside ampoule 10 is crucible 12. During operation, crucible 12 will contain seed crystal 14 in seed well 15. Crucible 12 will further contain Group II-VI or Group III-V compound 18. Ampoule 10 is positioned in furnace 17, said furnace 17 having a heating means comprised of upper heating component 18 and optionally lower heating component 19. Said heating means is mounted radially about at least a
 50 portion of said crucible 12 and is movable parallel to the vertical axis of said crucible 12. Preferably upper heating component 18 is comprised of two heating elements capable of individual temperature control and lower heating component 19 is comprised of two heating elements capable of individual temperature control. In the most preferred embodiment, a baffle 20 is located in furnace 17 between upper heating component 18 and lower heating component 19.

55 FIG. 2 is a detailed representation of a preferred embodiment of crucible 12. Crucible 12 is preferably constructed of pyrolytic boron nitride. With reference to FIG. 2, the lowest portion of crucible 12 is seed well 15 adapted for holding a seed crystal 14. Seed well 15 is an elongated member, preferably a small diameter cylinder having vertical walls. Moving up crucible 12, the next component is the cone-shaped

transition region 21. Transition region 21 allows the growing crystal to smoothly expand from the diameter of the seed to the final diameter of the mono-crystalline ingot from which semiconductor or semi-insulator wafers are cut. The angle of transition region 21 is about 45° . However, larger or smaller angles may also be employed.

Located above transition region 21 is primary growth chamber 22 which is also part of crucible 12. Most preferably, primary growth chamber 22 is cylindrical and has a diameter slightly greater than the diameter of the wafers to be cut from the monocrystalline semiconductor or semi-insulator ingot. The portion of the single crystal which solidifies in primary growth chamber 22 provides the useful material of the process, so it is desirable that primary growth region 22 have a substantial vertical length. In Fig. 2 the shaded parts 25 and 26 show the GaAs melt and solid crystalline GaAs, respectively. In a preferred embodiment of the current invention, crucible 12 is closed at its upper end with a cap means 23. In another preferred embodiment, opening 24 is provided in cap means 23 to allow fluid communication between the Group II-VI or Group III-V material inside crucible 12 and a source of carbon located outside crucible 12. In this context, "fluid communication" means a free flow of vapor and heat between the inside and outside of the crucible to enable transport of carbon into the crucible and to the melt. In a preferred embodiment, a disk of carbon 27 is placed over opening 24 on top of cap means 23. We have found that by placing about 0.7 cm^3 to about 2.3 cm^3 carbon outside crucible 12 and having an opening 24 of about 10 mm to about 30 mm diameter in cap 23, uniformly carbon-doped gallium arsenide ingots of about 50 mm to about 76 mm diameter and about 75 mm to about 150 mm long having a carbon level of about $0.5 \times 10^{15} \text{ cm}^{-3}$ to about $3.0 \times 10^{15} \text{ cm}^{-3}$ can be produced. However, ingots of other sizes and other doping levels may be produced by this technique by varying opening 24 and/or the amount of carbon placed outside crucible 12. For example, a gallium arsenide ingot of about 100 mm diameter, and produced by the technique within the scope of this invention, may require a larger opening 24 and/or more carbon than that within the ranges disclosed above.

In the process of the current invention, a powdered solid having a melting point higher than the melting point of the polycrystalline Group II-VI or Group III-V compound is applied to the interior surfaces of crucible 12. This may be accomplished by any reasonable means suitable to accomplish application of a powder to surfaces. For example, the powder may be dusted in dry form onto the interior surfaces of the crucible. In another embodiment a slurry of a carrier fluid and the powdered solid is prepared. The slurry is applied to the interior surfaces of the crucible and the liquid carrier is evaporated, leaving a deposit of powdered solid. Suitable liquid carriers may be, but are not limited to mixtures of water and alcohol. The preferred alcohol is methanol. Boron nitride is the preferred powdered solid when the Group III-V compound is gallium arsenide.

After coating crucible 12 with powdered solid, mono-crystalline seed 14 is placed in seed well 15 and polycrystalline material is placed in the remainder of crucible 12 (transition region 21 and primary growth region 22). Cap means 23 is placed on top of crucible 12, crucible 12 is placed into ampoule 10 (preferably constructed of quartz) which is then sealed by quartz plug 28 in a way which minimizes the free volume in the ampoule. The volatile Group V or Group VI component in an amount sufficient to fill the free volume may be added prior to sealing the ampoule. The sealed crucible-ampoule assembly is then placed on support means 13 in furnace 17. Furnace 17 is lowered and heating components 18 and 19 are employed so that all the polycrystalline material and as much as the top half of the monocrystalline seed are reduced to a melt. To grow a monocrystalline compound, furnace 17 is adjusted so that the temperature of upper heating component 18 is above the melting temperature of the Group III-V or Group II-VI compound and lower heating component 19 is below the melting point and there is a sharp temperature gradient of about $5\text{--}20^\circ \text{C/cm}$. across the transition zone between the upper hot zone and the lower cool zone. After adjusting furnace 17 to produce the above described temperature profile, furnace 17 is slowly moved up (about $1\text{--}8 \text{ mm/hr}$) parallel to the vertical axis of crucible 12 to produce a solid monocrystalline ingot. The ingot may be removed from the crucible and subjected to further processing, e.g. annealing, etc., then sliced into wafers for use, particularly in the electronics industry.

EXAMPLE 1

Semi-insulating gallium arsenide wafers were produced by the process of the current invention. A refined version of the apparatus illustrated in FIGS. 1 and 2 and described above was employed. Powdered boron nitride and a pyrolytic boron nitride crucible were individually heated, in the presence of flowing nitrogen, to 1100°C and held at that temperature for 2 hours, then cooled to room temperature. The interior of the crucible was dusted with the powdered boron nitride. A mono-crystalline seed crystal of gallium arsenide was placed into the crucible seed well and polycrystalline gallium arsenide was loaded into a

crucible having a 78 mm diameter x 200 mm length growth region. A small amount of arsenic in excess of that stoichiometrically required to combine with gallium was added to compensate for volatilization of arsenic during heating. There was no source of arsenic external to the crucible. The loaded crucible was placed in a sealed quartz ampoule and the ampoule-crucible assembly was placed on a rotating pedestal (rotation speed: 2 RPM). The hot zone (upper heating component) of the baffled furnace was set at 1243°C and the polycrystalline gallium arsenide and the upper half of the seed crystal were melted. The hot zone temperature was reduced at a rate of about 1°C/hour until the temperature reached 1240°C (2 degrees above the 1238°C melting point of gallium arsenide). The cold zone temperature was set to 1217°C, producing a temperature gradient across the transition zone of 5°C/cm. At this point, the furnace-lifting drive was started and monocrystalline growth began. Slower drive rates generally produce higher quality material, but the longer time required for growth increases costs. Here, the furnace was raised at about 4 mm/hour. When the entire ingot had been solidified, the temperature in both zones was reduced to 900°C at a rate of 50°C/hour. This rate is used to avoid thermal stress. The ingot is held at 900°C for 16 hours then the temperature is slowly lowered to room temperature. The ingot is easily removed from the crucible.

In the industry, a GaAs ingot is graded by the qualities of wafers cut from the last-to-freeze or "tail" portion of the ingot. The dislocation density (or EPD) of a wafer cut from the tail of the above-produced GaAs ingot was determined by the standard method of etching the wafers in molten KOH at 450°C for 40 minutes. The etch pits were then counted at 41 sites across the wafer. The median EPD and the range of EPD are reported in Table 1. For comparison, tail-portion data for wafers produced by others by the VGF and LEC methods are also reported at Table 1.

The EL2 of tail-portion wafers of the above-produced ingot was measured by infrared absorption. The results are also reported at Table 1 with comparison VGF and LEC data generated by others.

Table 1

	Electrical and Etch Pit Density (Dislocation Density) Data for GaAs			
	EPD (cm ⁻²)		EL2 (x 10 ¹⁶ cm ⁻³)	
	Median	Range	Median	Range
VB (Ex. 1)	2700	500 to 7800	0.95	0.85 to 1.1
VGF (Comp.) ¹	3200	800 to 18000	0.56	0.4 to 0.66
LEC (Comp.) ^{2,3,4}	70000	50000 to 100000	1.4	0.8 to 1.8
HEM (Comp.) ⁵	—	10000 to 20000	—	4.0 to 6.0

1. D.C. Look et al., "Uniformity of 3-in., Semi-Insulating Vertical-Gradient-Freeze GaAs Wafers", J. Appl. Phys. 66(2), 15 July 1989.

2. Litton Airtron GaAs Specification Sheet, 1985.

3. Showa Denko K.K. GaAs Substrate Specification, 22 Aug. 1988.

4. S. K. Brierley et al., "Correlation between Implant activation and EL2 in semi-insulating GaAs", 5th Conf. on Semi-insulating III-V Materials, Malmö, Sweden, 1988.

5. U.S. Patent 4 840 699, column 3, lines 49-64.

EXAMPLE 2

The resistivity, and thus the semi-insulating character, of monocrystalline GaAs can be increased by the controlled addition of dopants such as carbon.

In previously known methods for producing monocrystalline GaAs, such as LEC, the exposure of the GaAs melt to graphite parts of the furnace virtually insured the presence of carbon in the monocrystalline GaAs. The amount of carbon incorporated into LEC-produced GaAs may be controlled by varying the water content of the B₂O₃ encapsulant used in the process.

The monocrystalline compounds produced by the process of this invention may be grown in sealed quartz ampoules and thus are not exposed to graphite furnace parts. If carbon is desired in the monocrystalline compound, it must be added. Our new process for controllably adding carbon to a Group II-

VI or Group III-V monocrystalline compound is described in this Example 2.

Thirteen GaAs ingots were prepared by the method of Example 1 with the following modifications. After loading the polycrystalline GaAs and before placing the loaded crucible into the quartz ampoule, a cap (such as cap 23 in FIG. 1) was fitted on top of the crucible. The 80 mm diameter by 1 mm thick cap had a 10 mm diameter opening in its center as illustrated at FIG. 2. A 22 mm diameter x 2-3 mm thick carbon disk was placed on top of the opening. This crucible-carbon disk assembly was placed in the ampoule and the process described in Example 1 was then continued.

GaAs is deemed "semi-insulating" if the resistivity is greater than or equal to about 1×10^7 ohm-cm. Of the thirteen ingots manufactured by the method of this Example 2, all were semi-insulating at the ingot front and 10 were semi-insulating at the ingot tail.

EXAMPLE 3

The first 8 gallium arsenide ingots (A-H) listed on Table II were consecutively produced using a controlled carbon-doping process of the current invention. Specifically, the method described in Example 2 was used with the following modifications:

- (1) a 52 mm diameter growth region,
- (2) a 56 mm diameter by 1 mm cap,
- (3) a 30 mm diameter cap opening,
- (4) a 38 mm diameter by 2.5 mm thick carbon disk and
- (5) use of gallium oxide (0.1 gm/1000 gm GaAs) in the polycrystalline GaAs as a gettering agent.

The last 10 gallium arsenide ingots (I-R) listed on Table II were made by a process outside the current invention. To produce ingots I-R, the method used to produce ingots A-H was employed with the following modifications:

- (1) graphite powder (carbon) was added directly into the crucible with the polycrystalline GaAs, and
 - (2) there was no opening in the cap, obviating any need for a carbon source external to the crucible.
- As demonstrated by the data reported in Table II, the process of the instant invention provides surprisingly superior uniformity of carbon doping both within each gallium arsenide ingot and from ingot to ingot.

Table II

Carbon Concentration of Gallium Arsenide Ingots		
Ingot	Front ($\times 10^{18} \text{cm}^{-3}$)	Tail ($\times 10^{18} \text{cm}^{-3}$)
A	2,2	1,5
B	2,4	2,2
C	1,7	1,1
D	2,1	1,5
E	2,8	1,0
F	2,0	1,7
G	2,0	1,3
H	2,2	1,2
I	0,5	< 0,1*
J	1,5	< 0,1
K	0,1	< 0,1
L	4,0	0,8
M	0,9	0,5
N	1,1	< 0,1
O	1,2	0,2
P	1,2	0,5
Q	0,7	< 0,1
R	0,4	< 0,1

*The detection limit is $0,1 \times 10^{18} \text{cm}^{-3}$.

Claims

1. A process for producing monocrystalline Group II-VI or Group III-V compound from the polycrystalline form of said Group II-VI or Group III-V compound, said process comprising:
 - (a) coating the interior surface of a crucible with a powdered solid, said powdered solid having a melting point higher than the melting point of said polycrystalline form of said compound,
 - (b) placing an amount of Group II-VI or Group III-V polycrystalline compound into said coated crucible,
 - (c) placing said coated crucible containing said compound into a heating means,
 - (d) heating said coated crucible containing said compound to produce a melt of said compound within said crucible while maintaining said powdered solid in solid powdered form, and
 - (e) cooling said crucible and said compound to produce a monocrystalline compound.
2. The process of claim 1 wherein said powdered solid is boron nitride.
3. The process of claim 1 wherein said coating step (a) further comprises
 - (1) forming a slurry of said powdered solid and a liquid,
 - (2) applying said slurry to the interior surfaces of said crucible, and
 - (3) allowing said liquid to evaporate and thereby leave a deposit of powdered solid on the interior surfaces of said crucible.
4. The process of claim 1 wherein said Group II-VI or Group III-V compound is gallium arsenide.
5. A monocrystalline Group II-VI or Group III-V compound produced by the process of claim 1.
6. Gallium arsenide produced by the process of claim 1.
7. A semi-insulating material comprised of a monocrystalline gallium arsenide having an EL2 concentration between about $0,85 \times 10^{16} \text{cm}^{-3}$ and about $2,0 \times 10^{16} \text{cm}^{-3}$ and a dislocation density between about 500cm^{-2} and about 7800cm^{-2} .
8. A method for producing in a vertically-oriented crucible a monocrystalline Group II-VI or Group III-V material from the polycrystalline precursor of said monocrystalline material, said method comprised of:
 - (a) coating the interior surfaces of said crucible with a powdered solid having a melting point higher than the melting point of said polycrystalline precursor,

- (b) placing a monocrystalline seed in the bottom of said crucible,
- (c) loading the remainder of said crucible with said polycrystalline precursor,
- (d) placing said crucible in a vertically-oriented furnace, said furnace capable of producing an upper hot zone and a lower cool zone;
- 5 (e) adjusting said furnace position and said upper hot zone temperature to enable heating of said polycrystalline material to form a melt while about the lower half of said monocrystalline seed remains in solid form;
- (f) setting the temperature in said lower cool zone below the melting point of said monocrystalline material while maintaining the temperature in the upper hot zone above said melting point to establish a
- 10 solid-liquid interface, and
- (g) moving said furnace and said solid-liquid interface upward while substantially maintaining the temperature settings of step (f) to produce a solid monocrystalline material as said furnace and said solid-liquid interface move vertically upward.
- 9. The method of claim 8 wherein said monocrystalline Group II-VI or Group III-V material is gallium arsenide.
- 16 10. The method of claim 8 wherein said powdered solid is powdered boron nitride.
- 11. The method of claim 8 further comprising placing, prior to step (e), a carbon source inside said furnace yet outside said crucible, said carbon source being in fluid communication with said polycrystalline precursor.
- 20 12. A monocrystalline material produced by the method of claim 11.
- 13. A process for producing carbon-doped monocrystalline Group II-VI or Group III-V compound from the polycrystalline form of said Group II-VI or Group III-V compound, said process comprising:
 - (a) placing an amount of polycrystalline compound into a crucible, said polycrystalline compound selected from the group consisting of Group II-VI compounds and Group III-V compounds,
 - 25 (b) placing said crucible containing polycrystalline compound into a furnace,
 - (c) placing a carbon source inside said furnace and outside said crucible, said carbon source being in fluid communication with said polycrystalline compound,
 - (d) supplying heat from said furnace to said crucible and said polycrystalline compound to produce a melt of said compound within said crucible, and
 - 30 (e) reducing the temperature of said furnace to cool said crucible to produce a carbon-doped monocrystalline compound.
- 14. The method of claim 13 further comprising, prior to step (a), coating the interior surfaces of the crucible with a powdered solid having a melting point higher than the melting point of said Group II-VI or Group III-V compound.
- 35 15. The method of claim 14 wherein said powdered solid is powdered boron nitride.
- 16. The process of claim 13 wherein said Group II-VI or Group III-V compound is gallium arsenide.
- 17. A carbon-doped monocrystalline compound produced by the process of claim 13.
- 18. A process for producing carbon-doped monocrystalline Group II-VI or Group III-V compound from the polycrystalline form of said Group II-VI or Group III-V compound, said process comprising:
 - 40 (a) placing a monocrystalline seed into a crucible,
 - (b) placing an amount of polycrystalline compound into the crucible, said polycrystalline compound selected from the group consisting of Group II-VI compounds and Group III-V compounds,
 - (c) placing said crucible containing polycrystalline compound into a quartz ampoule,
 - (d) placing a carbon source inside said ampoule yet outside said crucible, said carbon source being in
 - 45 fluid communication with said polycrystalline compound,
 - (e) sealing said ampoule,
 - (f) placing said sealed ampoule into a furnace,
 - (g) increasing the temperature of said furnace to produce a melt of said polycrystalline compound, and
 - (h) decreasing the temperature of said furnace to cool said melt and produce a carbon-doped
 - 50 monocrystalline compound.
- 19. The process of claim 18 further comprising, prior to step (a), coating the interior surfaces of the crucible with a powdered solid having a melting point higher than the melting point of said Group II-VI or Group III-V compound.
- 20. The process of claim 19 wherein said powdered solid is powdered boron nitride.
- 55 21. The process of claim 20 wherein said Group II-VI or Group III-V compound is gallium arsenide.
- 22. A carbon-doped monocrystalline Group II-VI or Group III-V compound produced by the process of claim 18.
- 23. A semi-insulating material comprised of a monocrystalline gallium arsenide having an EL2 concentration

between about $0,85 \times 10^{16} \text{ cm}^{-3}$ and $1,5 \times 10^{16} \text{ cm}^{-3}$ and a dislocation density between about 500 cm^{-2} and 7800 cm^{-2} .

24. A semi-insulating material comprised of a monocrystalline gallium arsenide having an EL2 concentration between about $0,85 \times 10^{16} \text{ cm}^{-3}$ and $1,2 \times 10^{16} \text{ cm}^{-3}$ and a dislocation density between about 500 cm^{-2} and 7800 cm^{-2} .

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fig. 2

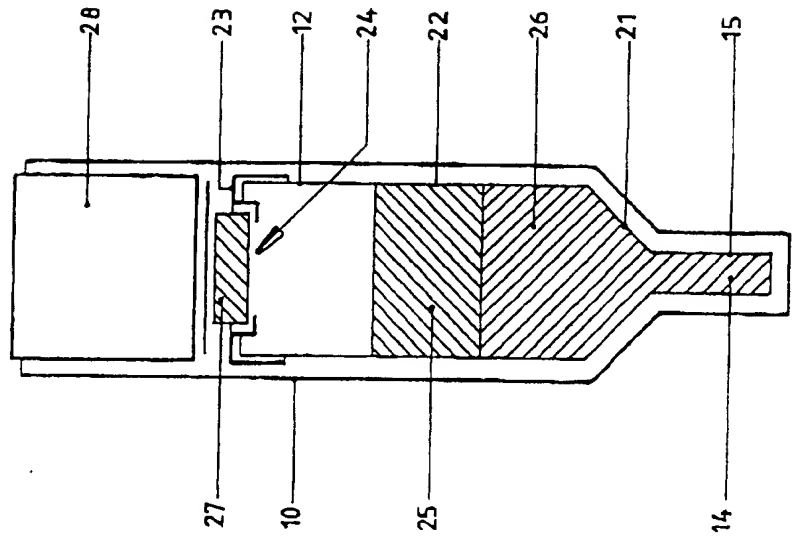


EXHIBIT 4: SECOND IDS

#7

Attorney's Docket No.: 12967-002001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Tomohiro Kawase et al Art Unit : 1765
Serial No. : 09/824,965 Examiner : Robert Kunemund
Filed : April 3, 2001
Title : METHOD OF PREPARING GROUP III-V COMPOUND SEMICONDUCTOR
CRYSTAL

Commissioner for Patents
Washington, D.C. 20231

SECOND INFORMATION DISCLOSURE STATEMENT

Applicant submits the references listed on the attached form PTO-1449, copies of which are enclosed.

Applicants have requested an interference with U.S. Patent No. 6,045,767 ("767 patent"). Items AA, AB and AO on the enclosed form PTO-1449 were cited in the prosecution of the '767 patent.

The following reference is listed on the face of the '767 patent:

"Semiconducting Gallium Arsenide [*sic.*] Single Crystals";
Yamamoto, et al., Japan. Kokai Tokkyo Koho, 5 pgs. (Abstract
only), 1989.

We believe that this is an incomplete reference to an abstract of Japan Patent Publication No. 64-37833, published Feb. 8, 1989, that is already cited as a reference in the '622 patent, which is the basis for the present reissue application.

Reference 803593A1, listed on the face of the '767 patent is the publication of the European counterpart to the present application.

Items AT, AU and AV on the enclosed form PTO-1449 are copies of documents which were found in the USPTO file of the '767 patent, but were not cited on the face of that patent.

An IDS was filed in the present reissue application by mail on or about December 14, 2001; however, a recent check of the file of this reissue application revealed that it had not been entered in the file. Items AL, AQ, AR and AS, identified and submitted with that first IDS are listed again on the enclosed form PTO-1449 and are being resubmitted at this time. Those items were brought to the undersigned's attention by Michael A. Molano, Esq., an attorney representing AXT Inc. (formerly American Xtal Technology), the assignee of the '767 patent.

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Applicant : Tomohiro Kawase et al
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 2

Attorney's Docket No.: 12967-002001

AXT contends that the three articles (items AQ, AR and AS) together show obviousness of one or more claims of the present application. Applicants disagree.

AXT also has suggested that reference AL, Kremer European patent publication EP 0,417,843 A2 ("Kremer EP"), is more relevant than Kremer, U.S. Patent No. 4,999,082 ("Kremer US"), which was cited in the prosecution of the parent of the present reissue application. (The claims of both SEI's '622 patent and AXT's '767 patent were allowed as patentably distinct from the Kremer U.S. patent.) AXT notes that the material under the heading "EXAMPLE 3" of Kremer EP does not appear in Kremer US. In particular, AXT contends:

[T]he Kremer EP publication shows boron oxide, solid carbon and that the two are related. Boron oxide in the form of B_2O_3 is disclosed in two parts of the Kremer EP publication: (a) p. 4, line 33 and (b) p. 7, line 55. Solid carbon is disclosed in Example 3 (p. 8, line 26). The two are "related" because:

At (b), the B_2O_3 is disclosed as part of Example 2 that, ... "is the same as the method" of Example 3.

The publication to B_2O_3 in Example 2 is immediately before the publication to "graphite powder (carbon)," separated only by about thirty (30) lines.

Example 3 specifically refers to Example 2 stating "the method described in Example 2 was used" with a number of modifications.

That position is wrong, for the reasons stated below. Kremer EP does not anticipate or make obvious any claims of SEI's '622 patent or its reissue application because it does not disclose, teach or suggest any of the claimed combinations.

1. Non-Anticipation by Reference AL (Kremer EP)

Kremer teaches the use of a high melting point powder, preferably boron nitride, to coat the crucible in the methods of his invention. In some preferred methods, Kremer provides carbon doping, using a carbon source within the sealed ampoule, separate from the crucible.

The method of EXAMPLE 1 discloses the use of boron nitride without carbon doping.

Applicant : Tomohiro Kawase et al
Serial No : 09/824,965
Filed : April 3, 2001
Page : 3

Attorney's Docket No.: 12967-002001

Two subjects are discussed under the heading EXAMPLE 2 in the Kremer EP publication. One is a previously known method, such as LEC using boron oxide, in which the carbon source is graphite parts of the furnace. (Page 7, lines 52-55) The other is Kremer's Example 2. The sealed ampoule, carbon doping method of Example 2 specifically incorporates by reference the method of Example 1. (Page 8, lines 6-7) It employs Kremer's carbon source separate from the crucible. Example 2 does not use boron oxide. In that Example, boron nitride or similar powder is used, per Example 1. (Page 7, line 56 - page 8, line 10) Persons skilled in the art would understand that no boron oxide would be used when boron nitride is used.

Two subjects also are discussed under the heading EXAMPLE 3 in the Kremer EP publication. First is Kremer's Example 3, a modified version of Example 2 (not of the LEC prior art) which also uses the separate carbon source within a sealed ampoule. (Page 8, lines 15-22) Finally, Kremer EP has a comparative example, "outside the current invention." The comparative example uses an unspecified amount of graphite powder (carbon) in the crucible. (Page 8, lines 23-30) It is a modification of Example 3 and, therefore, used boron nitride, not boron oxide.

The method of Kremer's Example 3 was used in making eight ingots A-H. (Page 8, lines 15-22). The comparative example was used for making ten ingots I-R. (Page 8, lines 26-27) Kremer EP goes on to discuss how the data in Table II demonstrates the superiority of the Kremer process over the comparative example process. (Page 8, line 28 - page 9, line 28)

Kremer EP does not disclose any of the claimed combinations.

For a prior art reference to anticipate a combination claim, every element must be identically shown in a single reference and must be arranged as in the claim under review. *See In re Bond*, 910 F.2d 831, 832 (Fed. Cir. 1990). The comparative example of Kremer EP, however, does not disclose the claimed combination of any of the claims of the reissue application. For example, while the comparative example does refer use of graphite powder (carbon), it does not disclose the claimed combination using B_2O_3 .

It would not be proper to combine two different parts of a publication to form an anticipatory reference unless the two parts are related. *See Ecolochem, Inc. v. Southern Cal. Edison Co.*, 227 F.3d 1361, 1368-69 (Fed. Cir. 2000). The comparative example of carbon powder-doped ingots I-R were made by a method similar to Kremer EP's Example 3, which in

Applicant : Tomohiro Kawase et al
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 4

Attorney's Docket No.: 12967-002001

turn states that it is the same as the method of Example 2, with certain, specified modifications. Example 2 also lacks any disclosure of B_2O_3 . While the prior art discussions in the Kremer US patent and Kremer EP application do refer to B_2O_3 , there is no statement in either reference disclosing, teaching or suggesting a combination including use of both B_2O_3 and carbon powder placed in the crucible. Therefore, that Kremer EP does not anticipate any claims of the reissue patent.

2. Non-Obviousness

Kremer EP also does not teach or suggest any of SEI's claimed combinations and, therefore, those claims are not obvious from Kremer EP under 35 U.S.C. § 103(a). Obviousness cannot be established by combining the teachings of the prior art to produce the claimed invention, absent some teaching or suggestion or motivation supporting the claimed combination. See *Ecolochem*, 227 F.3d at 1372-75. The claimed inventions of the present reissue application are not taught or suggested by the Kremer EP application, nor is there any motivation to combine the distinct and separate disclosures in that reference to make the claimed combinations of the reissue application.

References which teach away from the claimed combination also indicate the non-obviousness of that combination. See *Ecolochem*, 227 F.3d at 1373. The teaching of the Kremer EP publication, that the crucible should be coated with boron nitride powder (instead of using B_2O_3), teaches away from the combinations of the present reissue application claims. The criticism of using B_2O_3 in both Kremer references also teaches away from combining B_2O_3 with the carbon powder comparative example in Kremer EP. That confirms that Kremer EP does not incorporate use of B_2O_3 by reference into that comparative example and, therefore, that the combinations of the reissue claims are not obvious from Kremer EP.

The Kawase et al. Abstract and Article

Also submitted herewith are copies of two Kawase et al. documents, for the purpose of clarification. The Abstract (item AW) was published at the time of a conference, April 29 - May 3, 1996, and the longer paper (item AX) was published later in 1996. The paper was cited in the prosecution of the '622 and '767 patents. Neither is prior art with respect to the present reissue

Applicant : Tomohiro Kawase et al
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 5

Attorney's Docket No.: 12967-002001

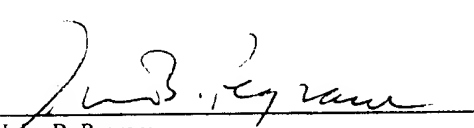
application because (1) it describes the present Applicants' work and (2) the present Applicants have claimed the priority of their earlier Japanese patent application 08-107009, filed April 26, 1996. A copy of that priority application and a translation are of record in the file of the '622 patent.

This statement is being filed before the receipt of a first Office action on the merits.

Please apply any charges or credits to Deposit Account No. 06-1050.

Respectfully submitted,

Date: January 26, 2002


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Substitute Form PTO-1449 (Modified)	U.S. Department of Commerce Patent and Trademark Office	Attorney's Docket No. 12967-002001	Application No. 09/824,965
Information Disclosure Statement by Applicant (Use several sheets if necessary)		Applicant Tomohiro Kawase et al	
		Filing Date April 3, 2001	Group Art Unit 1765

U.S. Patent Documents							
Examiner Initial	Desig. ID	Patent Number	Issue Date	Patentee	Class	Subclass	Filing Date If Appropriate
<i>[initials]</i>	AA	5,342,475	8/1994	Yoshida et al.	117/83		
	AB	5,454,346	10/1995	Uchida et al.	117/13		
	AC						
	AD						
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


Foreign Patent Documents or Published Foreign Patent Applications								
Examiner Initial	Desig. ID	Document Number	Publication Date	Country or Patent Office	Class	Subclass	Translation	
							Yes	No
<i>[initials]</i>	AL	0 417 843 A2	Mar. 20, 1991	EPO				
<i>[initials]</i>	AM	0529963B1	Aug. 21, 1992	EPO				
<i>[initials]</i>	AN	04-104989	Apr. 7, 1992	Japan			Abs	
<i>[initials]</i>	AO	06-128096	May 10, 1994	Japan			Abs	
	AP							

Other Documents (include Author, Title, Date, and Place of Publication)		
Examiner Initial	Desig. ID	Document
<i>[initials]</i>	AQ	Parsey, "Relative virtues of different growth techniques" in Semi-Insulating III-V Materials, 1988.
<i>[initials]</i>	AR	Doering et al., "Carbon incorporation into LEC GaAs" in Semi-Insulating III-V Materials, 1990.
<i>[initials]</i>	AS	Müller et al., "Current issues in bulk growth of s.i. III-V materials" in Semi-Insulating III-V Materials, 1992.
<i>[initials]</i>	AT	Doering et al., "Carbon Incorporation Into LEC GaAs, Int. Conf. Semi-conducting and Semi-Insulating GaAs," Malmö, Sweden (1984)
<i>[initials]</i>	AU	Desnica et al., "Distribution coefficient of carbon in gallium arsenide," Inst. Phys. Conf. Ser. No. 83, ch. 2, pp. 33-38 (1986)

Examiner Signature <i>[Signature]</i>	Date Considered 3/4/02
EXAMINER: Initials citation considered. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.	

Sheet 2 of 2

Substitute Form PTO-1449 (Modified)	U.S. Department of Commerce Patent and Trademark Office	Attorney's Docket No. 12967-002001	Application No. 09/824,965
Information Disclosure Statement by Applicant (Use several sheets if necessary) (37 CFR §1.98(b))		Applicant Tomohiro Kawase et al	
		Filing Date April 3, 2001	Group Art Unit 1765

Other Documents (include Author, Title, Date, and Place of Publication)		
Examiner Initial	Desig. ID	Document
	AV	Desnica et al., "Distribution coefficient of carbon in melt-grown GaAs," J. Appl. Phys. 62(9) (1 Nov. 1987)
	AW	Kawase et al., "Low-dislocation-density and Low residual-strain Semi-insulating GaAs Grown by VB Method," 9 th Conf. on Semiconducting & Insulating Mat'ls-Abstracts, title, contents and p. 47 (April 29-May 3, 1996)
	AX	Kawase et al., "Low-dislocation-density and Low-residual-strain Semi-insulating GaAs Grown by Vertical Boat Method," 9 th Conf. on Semiconducting & Insulating Mat'ls-Proceedings, title, pp. ii-iii, vii-viii, xv, 275-278 (1996)

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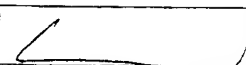
Examiner Signature 	Date Considered 3/4/02
EXAMINER: Initials citation considered. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.	

EXHIBIT 5: ECOLOCHEM CASE

1990). When the registration does not contain limitations describing a particular channel of trade or class of customer, the goods or services are assumed to travel in all normal channels of trade. *See Canadian Imperial Bank of Commerce v. Wells Fargo Bank, N.A.*, 811 F.2d 1490, 1492, 1 USPQ2d 1813, 1814-15 (Fed.Cir.1987). The Board properly based its analysis on Packard's registration, which contains no restrictions as to a particular class of consumer or channel of trade, and assumed that applicant and opposer's goods and services may be marketed "in some of the same manners" to the same classes of purchasers. *Hewlett-Packard*, slip op. at 8, 1999 WL 792477. We perceive no reversible error in the Board's treatment of these *DuPont* factors.

CONCLUSION

For the reasons stated above, we vacate the Board's decision and remand the case to the Board for further proceedings consistent with this decision.

COSTS

No costs.

VACATED AND REMANDED



ECOLOCHEM, INC., Plaintiff-Appellant,

v.

SOUTHERN CALIFORNIA EDISON COMPANY, Defendant-Appellee.

No. 99-1043.

United States Court of Appeals,
Federal Circuit.

Sept. 7, 2000.

Patentee of deoxygenation processes for liquid containing dissolved oxygen brought patent infringement action against operator of nuclear generating station. Op-

erator counterclaimed for declaratory judgment of invalidity, and asserted equitable defenses. The United States District Court for the Central District of California, 863 F.Supp. 1165, granted partial summary judgment for operator, invalidating certain claims. The Court of Appeals affirmed in part and reversed in part. Following bench trial, the District Court, Richard A. Paez, J., 1998 WL 1182000, found willful infringement, but determined that all infringed claims were invalid. Patentee appealed. The Court of Appeals, Michel, Circuit Judge, held that: (1) patent claims were not anticipated by prior art articles; (2) one claim was anticipated by scientist's public presentation; (3) majority of claims were not rendered obvious by prior art, absent motivation to combine prior art references; but (4) patentee did not rebut prima facie case of obviousness as to one claim.

Affirmed in part, reversed in part, and remanded.

1. Patents \Rightarrow 70

Claims in patent for deoxygenation processes for liquid containing dissolved oxygen were not anticipated by prior art articles, since articles' discussion of applications of deoxygenated water was limited to use of hydrogen deoxygenation, while patent claims involved deoxygenation by hydrazine in combination with a mixed bed ion exchange resin. 35 U.S.C.A. § 102(a).

2. Patents \Rightarrow 324.55(4)

Court of Appeals reviews for clear error the district court's decision on anticipation of a patent claim after trial. 35 U.S.C.A. § 102(a).

3. Patents \Rightarrow 60

Claims in patent for deoxygenation processes for liquid containing dissolved oxygen, which required removal of excess hydrazine in the final ion exchange step, were not anticipated by scientist's public presentation at International Water Conference, which did not discuss removal of

excess hydrazine, but presentation did anticipate claim directed to deoxygenation process comprising steps of passing liquid containing oxygen and hydrazine through activated carbon, and then passing liquid through mixed bed ion exchange resins to remove at least the dissolved contaminants. 35 U.S.C.A. § 102(a).

4. Patents ⇨60

A public presentation indicative of the state of knowledge and use of an invention in the United States qualifies as prior art for anticipation purposes under the patent statute. 35 U.S.C.A. § 102(a).

5. Patents ⇨324.55(4)

Court of Appeals reviews de novo the district court's conclusion of obviousness of a patent claim. 35 U.S.C.A. § 103(a).

6. Patents ⇨16.5(1)

Patent claims directed to deoxygenation processes for liquid containing dissolved oxygen, which used process set forth in prior art article, including use of carbon bed, followed by use of mixed bed ion exchange resin downstream of carbon bed, were not rendered obvious by combination of article and other references, as there was no evidence of any suggestion, teaching, or motivation to combine article and other references, and there was evidence of teaching away from demineralization and deoxygenation processes used by patentee. 35 U.S.C.A. § 103(a).

7. Patents ⇨16(4)

Court of Appeals cannot use hindsight reconstruction to pick and choose among isolated disclosures in the prior art to deprecate the claimed invention pursuant to a claim that patent is invalid for obviousness. 35 U.S.C.A. § 103(a).

8. Patents ⇨26(1)

When a rejection for obviousness depends on a combination of prior art references, there must be some teaching, suggestion, or motivation to combine the references, and the same principle applies to invalidation of the patent. 35 U.S.C.A. § 103(a).

9. Patents ⇨16(4), 26(1)

Although the suggestion to combine prior art references may flow from the nature of the problem, defining the problem in terms of its solution reveals improper hindsight in the selection of the prior art relevant to obviousness; therefore, when determining the patentability of a claimed invention which combines two known elements, the question is whether there is something in the prior art as a whole to suggest the desirability, and thus the obviousness, of making the combination. 35 U.S.C.A. § 103(a).

10. Patents ⇨36(1)

Broad conclusory statements regarding the teaching of multiple prior art references, standing alone, are not evidence of a motivation to combine those references, as would support a claim of obviousness. 35 U.S.C.A. § 103(a).

11. Patents ⇨36.1(2, 3), 36.2(1)

Patentee failed to rebut prima facie case of obviousness as to claim for deoxygenation process for liquid containing dissolved oxygen, despite some evidence of secondary considerations of nonobviousness such as modest commercial success and teaching away from use of prior art process incorporated in claimed process, in light of absence of any evidence that others were trying to emulate patented process, and fact that patentee applied for patent on the process within two years of publication of industry guidelines recommending use of deoxygenated water in nuclear power plants. 35 U.S.C.A. § 103(a).

12. Patents ⇨324.5, 324.55(4)

While Court of Appeals review the district court's factual findings on the secondary considerations of obviousness for clear error, on claim of patent invalidity, Court reviews the ultimate determination of obviousness de novo. 35 U.S.C.A. § 103(a).

13. Patents ⇨36.2(4), 324.55(4)

District court clearly erred in finding that operator of nuclear generating station

established the invention, name for liquid control due to factors invention, for claim was inventor's own was based both embodiment of claimed, and operation process, U.S.C.A. § 103.

14. Federal Circuit

Court of Appeals to the district court regarding credibility.

15. Patents ⇨

The fact of invention, though not obviousness, is constitutes the legal art, although the taneous invention talented inventor may or may not obviousness when certain circumstances. 3

16. Patents ⇨36

District court that prior art disclosing process set or from combining exchange resin in claimed deoxygenation process used in prior art, for purpose of used to deoxygenation in view of various against use of prior carbon contaminant in prior art process expensive. 35 U.S.C.A. § 103(a).

17. Patents ⇨36.1

One indicia of patented product is receives when it is in that occurs. 35

18. Patents ⇨36.1

Court's belief that was not novel was

established that success of patentee's invention, namely, a deoxygenation process for liquid containing dissolved oxygen, was due to factors not claimed in the patented invention, for purpose of claim that patent claim was invalid as obvious, in light of operator's own statements that success was based both on mobility of commercial embodiment of invention, which was not claimed, and on invention's improved filtration process, which was claimed. 35 U.S.C.A. § 103(a).

14. Federal Courts ⇨844

Court of Appeals gives great deference to the district court's decisions regarding credibility of witnesses.

15. Patents ⇨34

The fact of near-simultaneous invention, though not determinative of statutory obviousness, is strong evidence of what constitutes the level of ordinary skill in the art, although the possibility of near simultaneous invention by two or more equally talented inventors working independently may or may not be an indication of obviousness when considered in light of all the circumstances. 35 U.S.C.A. § 103(a).

16. Patents ⇨36(3)

District court clearly erred in finding that prior art did not teach away from using process set forth in prior art article, or from combining it with a mixed bed ion exchange resin in any manner, to develop claimed deoxygenation and demineralization process used in nuclear power generator, for purpose of claim that patent directed to deoxygenation process was obvious, in view of various references warning against use of prior art process due to carbon contaminants, and reference finding prior art process to be inefficient and expensive. 35 U.S.C.A. § 103(a).

17. Patents ⇨36.1(2, 5)

One indicia of non-obviousness of a patented product is the acclamations it receives when it is released, and the copying that occurs. 35 U.S.C.A. § 103(a).

18. Patents ⇨36.1(2)

Court's belief that patented process was not novel was not proper basis for

discounting copying of claimed invention as evidence of nonobviousness. 35 U.S.C.A. § 103(a).

19. Patents ⇨36.1(2)

A showing of copying of a claimed invention is only equivocal evidence of non-obviousness in the absence of more compelling objective indicia of other secondary considerations, because the alleged copying could have occurred out of a general lack of concern for patent property. 35 U.S.C.A. § 103(a).

Clifton E. McCann, Lane, Aitken & McCann, of Washington, DC, argued for plaintiff-appellant. With him on the brief was Andrew C. Aitken.

Ted G. Dane, Munger, Tolles & Olson, of Los Angeles, California, argued for defendant-appellee. With him on the brief was Gregory P. Stone.

Before MICHEL, CLEVINGER, and RADER, Circuit Judges.

MICHEL, Circuit Judge.

Ecolochem, Inc. ("Ecolochem") filed suit in 1992, alleging that Southern California Edison Company ("Edison") infringed Ecolochem's U.S. Patent Nos. 4,556,492 ("the '492 patent") and 4,818,411 ("the '411 patent") when deoxygenating water in the High-Flow Makeup Demineralizer ("HFMUD") at Edison's San Onofre Nuclear Generating Station ("SONGS"). Edison denied infringement, counterclaimed for declaratory judgment of invalidity, and asserted equitable defenses. By grant of partial summary judgment to Edison, the United States District Court for the Central District of California invalidated claims 1, 2, and 5-10 of the '492 patent and claims 20-21 of the '411 patent, holding the subject matter of each of these claims to be either anticipated under 35 U.S.C. § 102 and/or obvious under 35 U.S.C. § 103. On appeal to this court, we reversed the holding by the district court that there was no genuine issue of material

fact that the invention of claim 20 of the '411 patent would have been obvious at the time of the invention, and remanded the case for a trial on invalidity in light of Ecolochem's evidence of secondary considerations. As to the invalidation of the other appealed claims, we affirmed. Ecolochem continued to assert infringement of claims 1, 3-13, 15, 17, 18, and 20 of the '411 patent after remand, but dropped its suit as to the remaining claims of the '492 patent. After a bench trial, the district court found that Edison had willfully infringed claims 1, 3-13, 15, 17, 18, and 20 of Ecolochem's '411 patent and rejected Edison's equitable defenses. The court then went on to invalidate all of the claims found to be infringed. Ecolochem appeals the holdings of invalidity as to claims 1, 3-13, 15, 17, 18, and 20 of the '411 patent here. Edison does not cross-appeal the district court's finding that Edison willfully infringed those claims of the '411 patent.

We affirm the district court's finding that claim 20 of the '411 patent was proven invalid by clear and convincing evidence both as anticipated under 35 U.S.C. § 102 and obvious under 35 U.S.C. § 103. We reverse its findings of invalidity for anticipation for claims 1, 4, and 7-12 because we discern clear error in the district court's finding that the prior art was proven by clear and convincing evidence to have recited every limitation of claims 1, 4, and 7-12. We also reverse the district court's conclusions that the subject matter of claims 1, 3-13, 15, 17, and 18 of the '411 patent was proven invalid for obviousness by clear and convincing evidence, as we discern clear error in the district court's implicit finding that there was motivation to combine the teachings of the prior art references. As the district court's findings of willful infringement stand unchallenged, we remand for a determination of damages.

BACKGROUND

Edison operates SONGS, a type of nuclear power plant known as a Pressurized Water Reactor ("PWR"). PWRs use water in two systems, called the "primary

system" and the "secondary system." A minute amount of water from the secondary system is lost during each operation cycle. This water must be replaced, and the replacement water is commonly referred to as "make-up water." Because water in the secondary system must be of extremely high purity, make-up water is supplied from a make-up demineralization system, which takes outside source water and refines it through "demineralization," i.e., the removal of mineral ions.

During the start-up operations for SONGS, Edison decided to construct a HFMUD to meet the make-up water needs of SONGS' two active reactors. While the HFMUD was being constructed, Edison employed outside vendors, including Ecolochem, to provide the needed make-up water. Before hiring Ecolochem, Edison hired other vendors who provided poor quality water and constantly shuttled demineralization trucks on and off Edison's property to meet Edison's requirements. In the summer of 1982, Edison hired Ecolochem to provide purer quality water at the large volumes Edison needed to meet its make-up water needs. Ecolochem used a patented "Mobile Flow" trailer apparatus to provide water treatment services, which enabled it to regenerate its trailers on-site, thus avoiding the other vendors' needs to truck the impure water off-site. Ecolochem, however, was hired only to produce demineralized water, not water that had been both demineralized and deoxygenated.

The Electrical Power Research Institute ("EPRI"), a research organization for the power industry, published new guidelines in 1982, recommending the use of deoxygenated water in PWRs. These guidelines were soon implemented throughout the utility industry. In direct response to these guidelines, Edison asked Ecolochem to deoxygenate the make-up water used in SONGS. Shortly thereafter, Ecolochem began developing the patented process at issue in the instant case.

Once constructed and finished, Edison Ecolochem's service produces, on its own, deoxygenated make-up water through the water through HFMUD passes through a strong cation bed, predominantly water bed, an activated strong acid cation anion bed, and, finally, a filter. This process removes dissolved solids and impurities, including salt, minerals, and oxygen.

Ecolochem alleges that Edison's process, as described in its patent, Ecolochem's dependent claims (multiple dependent claims read as

1. A deoxygenated water, as a first step of contacting [sic] dissolved oxygen with a bed of catalyst, thereby catalyze a reaction between dissolved oxygen and carbon contaminants, whereby said contaminants are removed from said liquid through a change resin and exchange resin.

15. A deoxygenated water, as a first step of contacting dissolved oxygen with a bed of activated carbon, whereby an amount of dissolved activated carbon are added to said water.

1. Cation: An ion having a positive charge. See Webster's Ninth New International Dictionary 216 (1990).

2. Anion: An ion having a negative charge. See Webster's Ninth New International Dictionary 87 (1990).

Once construction of the HFMUD was finished, Edison had no further need for Ecolochem's services, being able to now produce, on its own, sufficient, high purity, deoxygenated make-up water by passing the water through the HFMUD. The HFMUD passes the make-up water through a strong acid cation¹ resin bed, a predominantly weak base anion² resin bed, an activated carbon bed, a second strong acid cation resin bed, a strong base anion bed, and, finally, a vacuum deaerator. This process removes suspended, undissolved solids and dissolved impurities, including salt, mineral ions, organic chemicals, and oxygen.

Ecolochem alleged that Edison's process, as described above, infringes its '411 patent. Ecolochem asserted all three independent claims of the '411 patent, and multiple dependent claims. The independent claims read as follows:

1. A deoxygenation process comprising a first step of contacting a liquid contacting [sic] dissolved oxygen and hydrazine with a bed of activated carbon to catalyze a reaction between said dissolved oxygen and a portion of said hydrazine, whereby an amount of dissolved carbon contaminants is added to said liquid, and a second step of removing said contaminants and said unreacted hydrazine that comprises passing said liquid through a strong acid cation exchange resin and a strong base anion exchange resin.

15. A deoxygenation process comprising a first step of contacting water containing dissolved oxygen and hydrazine with a bed of activated carbon to catalyze a reaction between said dissolved oxygen and a portion of said hydrazine, whereby an amount of dissolved and undissolved activated carbon contaminants are added to said water, a second step of

removing said dissolved contaminants and said unreacted hydrazine by passing said water through a strong acid cation exchange resin and a strong base anion exchange resin, said water being at a temperature above the freezing point of water and below a temperature that would damage said resins, removing said undissolved contaminants by passing said water through a filter whereby said undissolved contaminants are filtered from said water, and a fourth step of circulating said water in a power generating apparatus after said removing step.

20. A deoxygenation process comprising a first step of contacting a liquid containing dissolved oxygen and hydrazine with a bed of activated carbon to catalyze a reaction between said dissolved oxygen and said hydrazine, whereby an amount of dissolved activated carbon contaminants is added to said liquid, and a second step of removing at least said dissolved contaminants by passing said liquid through a strong acid cation exchange resin and a strong base anion exchange resin.

Asserted claims 3-13 depend on claim 1. Asserted claims 17 and 18 depend on claim 15. Claim 20 is independent, and no claims dependent thereon are asserted.

All asserted claims in suit recite a deoxygenation process for removing dissolved oxygen from a liquid. In representative claim 20, liquid containing oxygen and hydrazine passes through activated carbon, thus catalyzing a reaction between the oxygen and hydrazine, whereby an amount of dissolved carbon contaminants falls out of the reaction and is added to the liquid. Then the liquid passes through ion exchange resins, including both strong acid cation and strong base anion exchange resins,³ to remove at least the dissolved con-

1. Cation: An ion having a positive charge. See Webster's Ninth New Collegiate Dictionary 216 (1990).

2. Anion: An ion having a negative charge. See Webster's Ninth New Collegiate Dictionary 87 (1990).

3. A cation/anion exchange resin is a resin which, when brought in contact with liquid containing cation/anion contaminants, removes them.

taminants. The invention of independent claim 1 is similar, except that the resins also remove any unreacted hydrazine. Claims 3 through 6 and claims 10 through 13 require further steps in the deoxygenation process, e.g., filtration. Claim 7 requires the temperature of the liquid to be above the liquid's freezing point, and below the temperature at which the resins would be damaged. Claim 8 requires the liquid to be water. Claim 9 requires that the liquid of claim 1 be water and that the water be circulated in a power generating apparatus. Independent claim 15 recites a combination similar to claim 9, and claims 17 and 18 add hydrazine and demineralize the water.

The '411 patent issued in 1989 from a continuation of an application which issued as the '492 patent on December 5, 1985. Both the '411 and '492 patents are entitled "Deoxygenation Process." At the outset of this litigation, Ecolochem asserted claims under both the '492 and '411 patents.

On September 1, 1994, Judge Gadbois, the original trial judge, granted-in-part and denied-in-part Edison's motion for summary judgment seeking to invalidate various asserted claims of Ecolochem's patents. See *Ecolochem, Inc. v. Southern Cal. Edison Co.*, 863 F.Supp. 1165 (C.D.Cal.1994). Judge Gadbois held claims 1, 2, and 5-10 of the '492 patent and claims 20 and 21 of the '411 patent to be invalid as either obvious and/or anticipated. Ecolochem appealed the partial summary judgment as to claims 1, 2, 5-7, and 10 of the '492 patent and claim 20 of the '411 patent. Ecolochem did not appeal the findings of invalidity of claims 8 and 9 of the '492 patent or claim 21 of the '411 patent. We affirmed the district court's holdings of anticipation of claims 1, 2, 5, and 6 and obviousness of claims 7 and 10 of the '492 patent. We reversed the summary judgment of obviousness of claim 20 of the '411 patent and remanded the case for a trial weighing the secondary consid-

eration evidence as to claim 20. See *Ecolochem, Inc. v. Southern Cal. Edison Co.*, No. 95-1320, 91 F.3d 169, 1996 WL 297601 (Fed. Cir. June 5, 1996) (table).

Upon remand, the case was randomly reassigned to Judge (now Circuit Judge) Richard A. Paez⁴ to conduct a trial on claim 20 and the remaining claims upon which summary judgment had not been granted in the earlier district court decision. Ecolochem asserted infringement with respect to claims 1, 3-13, 15, 17, 18, and 20 of the '411 patent. See *Ecolochem, Inc. v. Southern Cal. Edison*, No. 92-3436, 1998 WL 1182000, *5 (C.D.Cal.1998) ("*Ecolochem*"). After a bench trial, Judge Paez found that Edison had willfully infringed claims 1, 3-13, 15, 17, 18, and 20 of Ecolochem's '411 patent. Judge Paez, however, held claims 1, 3-13, 15, 17, 18, and 20 of the '411 patent to be invalid for obviousness and claims 1, 4, 7-12 and 20 also to be invalid as anticipated.

Ecolochem appeals the rulings of the district court that claims 1, 4, 7-12, and 20 were anticipated and that the inventions of claims 1, 3-13, 15, 17, 18, and 20 would have been obvious. Edison does not cross-appeal the district court's findings of willful infringement.

We have jurisdiction under 28 U.S.C. § 1295(a)(1) (1994).

ANALYSIS

I. Anticipation

A. The Martinola Reference

[1, 2] The district court found claims 1, 4, 7-12, and 20 of the '411 patent to be anticipated by either of two articles, one published in 1980 and the other in 1981, by Dr. Friedrich Martinola and a co-author, both of which are entitled "Saving Energy by Catalytic Reduction of Oxygen in Feedwater." See Dr. Friedrich Martinola & P. Thomas, *Saving Energy by Catalytic Reduction of Oxygen in Feedwater*, in *Pro-*

the United States Court of Appeals for the Ninth Circuit.

4. On March 9, 2000, the Senate confirmed the nomination of Judge Richard A. Paez to

ceedings of Conference Friedrich M ing Energy ygen in Feed Treatment J Because the aspects ident er throughout ence." We r sion on anti error. See 1 GMBH v. A Co., 730 F.2d 488 (Fed.Cir.1

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20. See *Ecol. Edison Co.*, 96 WL 297601 (E).

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ceedings of the 41st International Water Conference Pittsburgh 77 (1980); Dr. Friedrich Martinola and P. Thomas, *Saving Energy by Catalytic Reduction of Oxygen in Feedwater, in Effluent and Water Treatment Journal* 542 (December 1981).⁵ Because these two articles are in most aspects identical, we refer to them together throughout as "the Martinola reference." We review the district court's decision on anticipation after trial for clear error. See *Lindemann Maschinenfabrik GMBH v. American Hoist and Derrick Co.*, 730 F.2d 1452, 1462, 221 USPQ 481, 488 (Fed.Cir.1984).

The district court focused on Figure 10 of the Martinola reference (which is the same figure in both articles). Figure 10 shows a few possible uses for deoxygenated water, such as supplying make-up water for boilers, or for nuclear reactors. The figure is essentially a flow chart indicating various ways for the water to be deoxygenated, depending on the potential destination of the water. Water designated for nuclear reactors is shown flowing through three steps: demineralization, and then a two-step deoxygenation process, which is accomplished by passing the water through a catalyst column and a mixed bed.⁶ The district court interpreted Figure 10 as showing that the catalyst column can contain either the Lewatit catalyst or activated carbon. The district court found that the reactant can be either hydrogen or hydrazine.

On the face of the 1980 Martinola article, which fails to provide a detailed description of Figure 10, there is a bold heading reading: **Application of oxygen reduction in water with hydrogen.** Beneath this heading are two paragraphs of text, followed by Figure 10. The text reads:

The process is recommended wherever the heat used for thermal degassing cannot be recovered. It will therefore be

primarily used where the work has to be carried out at ambient temperature or where there is no heating steam available.

Fig. 10 shows a few suggested applications. It also includes systems for the simultaneous treatment of water with ion exchange resins and oxygen reduction with catalysts.

Martinola at 81-82 (1980). The 1981 article, on the other hand, has no bold headings, but discusses Figure 10 in more detail. The paragraph directly following Figure 10 reads:

[T]he application of hydrogen to reduce oxygen in water is recommended wherever the heat used for thermal degassing cannot be recovered. It will therefore be primarily used where the work has to be carried out at ambient temperature or where there is no heating steam available.

Figure 10 shows a few suggested applications.

Martinola at 546 (1981). The latter description clarifies that Figure 10 is not meant to illustrate the use of either hydrogen or hydrazine, but only hydrogen, contrary to the findings of the district court in the instant case. This is particularly apparent when we examine the differences between the two articles. The heading in the 1980 article clearly illustrates that Figure 10 refers only to hydrogen, and the discussion in the text explains only that Figure 10 shows a few applications. The 1981 article does not limit Figure 10's applications to hydrogen through a heading, but states clearly in the text ("the application of hydrogen to reduce oxygen.... Figure 10 shows a few suggested applications.") that the suggested applications are for the utilization of hydrogen to reduce oxygen in water.

5. The International Water Conference is an annual convention focusing on water treatment issues.

6. A mixed bed is a resin bed which, when brought in contact with liquid containing cation and/or anion contaminants, removes them. In other words, a resin bed containing a mixture of cation and anion resins.

We hold that the district court clearly erred in finding that the articles anticipate claims 1, 4, 7-12, and 20 of the '411 patent. Each article is entitled "Saving Energy by Catalytic Reduction of Oxygen in Feedwater." Each article discusses methods to deoxygenate water, dividing the methods in use at the time of the article into physical processes (vacuum degassing at low temperatures and pressure degassing at high temperatures), and chemical processes (reduction with sulfite, hydrazine or hydrogen). The articles state that the physical processes (or thermal degassing) have been the most common method of deoxygenating, as the chemical processes are expensive, slow to react at low temperatures, and contaminate the water by adding salts. The articles then state that the chemical process of deoxygenating water with hydrazine had been in use, but that the chemical process of deoxygenating water with hydrogen had not been, and concludes that the hydrogen process is preferable.⁷ At the end of the articles is a comparison of the thermal degassing method, the hydrazine method, and the hydrogen method. The articles state that:

Precondition for the proper use of hydrazine is the pH-value of the water to be higher than 8.5, because only in this range the reaction with oxygen takes place with sufficient rate. . . . When applying activated carbon as a catalyst in the removal of oxygen with hydrazine at ambient temperatures it has to be taken into account that the carbon releases salts into the demineralized water.

Martinola at 81 (1980); Martinola at 545-46 (1981). The articles then conclude that "[i]f we compare the final costs for all three processes . . . we find that the method of oxygen reduction with hydrogen is much cheaper than the other methods. The required apparatus is also simple and

needs virtually no maintenance." Martinola at 81 (1980); Martinola at 546 (1981). This conclusion is followed by a discussion of how to use water deoxygenated by hydrogen in different industries, as illustrated by Figure 10. Only in that discussion is there a suggestion to follow the catalyst column with a mixed bed if one plans to use water deoxygenated by hydrogen in nuclear power plants. The articles do not discuss the use of water deoxygenated by any other method in nuclear power plants. The articles' discussion of applications of deoxygenated water is limited to the use of hydrogen deoxygenation.

The district court in the instant case disagreed with the earlier discussion of the Martinola reference by the district court in *Ecologchem, Inc. v. Mobile Water Tech. Co.*, 690 F.Supp. 778 (E.D.Ark.1988), *aff'd*, 871 F.2d 1096, 10 USPQ2d 1557, 1989 WL 16031 (Fed.Cir.1989) (table). In the earlier case, the district court held that:

[t]he diagram found on page 82 of the Martinola article is under the bold-faced heading "Application of oxygen reduction in water with hydrogen." [See Martinola at 81 (1980)] (emphasis supplied). Thus, the diagram refers to applications of the process the authors are trying to promote—the palladium/hydrogen catalysis—and not the hydrazine process which is discussed in a separate section. This conclusion is buttressed by the fact that the palladium/hydrogen process may also release ionic impurities into the effluent, *see id.* at 79 ("traces of chlorides or other ions may be released"), thus necessitating a downstream ion exchange resin when high purity deoxygenated water is required. . . . Nothing in the Martinola reference expressly teaches the use of a mixed bed ion ex-

hydrazine were used in the final stage of deoxygenation, after the thermal degassing or other chemical process had been used, to remove the last traces of oxygen, while keeping to a minimum the contaminate byproducts of the hydrazine process.

7. The '411 specification explains further that "[i]n the prior art deoxygenation [sic] processes, hydrazine has been used as a strong reducing agent to prevent corrosion and other problems associated with oxygenated water." '411 pat., col. 1, ll. 27-30. A review of the prior art indicates that small amounts of

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Id. at 781-82.
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Id. at 781-82. This is to be contrasted with the district court in the instant case, which wrote:

[T]he Court respectfully disagrees with the district court's conclusion in *Mobile Water* that because Diagram 10 is positioned on the page following the heading "Application of oxygen reduction in water with hydrogen" and the associated text, Diagram 10 necessarily refers exclusively to reduction of oxygen in water with hydrogen and not to reduction of oxygen with hydrazine in an activated carbon column.... There is no room for Figure 10 at the bottom of page 81 because Figure 9 extends too far down the page. Following Figure 9 is a brief conclusion to the article, in which the authors recommend use of the Martinola system whenever deoxygenation is to be carried out at ambient temperatures.... Thus, Figure 10 refers generally to methods of deoxygenating and purifying water.

Ecologychem, at *38.

The district court clearly erred by misconstruing Figure 10's relationship to the text of the article. As discussed above, this becomes even clearer once we examine the difference between the two articles. Dr. Martinola and his co-author very carefully made sure in both the 1980 article, through the heading, and the 1981 article, through the language, that Figure 10 refers only to hydrogen and not to hydrazine. The authors would not have been so careful with the language in the 1981 article if they had not meant for Figure 10 to come under the "hydrogen-only" heading in the 1980 article. We disagree with the district court in this case that the publishers rather than the authors chose where to place Figure 10. Rather, we agree with the Arkansas district court that the authors intended the placement of Figure 10 under the hydrogen-only heading, and that Figure 10 therefore only refers to applications of the palladium/hydrogen catalysis. The district court, we hold, clearly erred in finding that Martinola anticipates deoxy-

genation by hydrazine in combination with a mixed bed. Consequently, we reverse the district court's finding that the Martinola reference was proven by clear and convincing evidence to have anticipated certain of the asserted claims of the '411 patent.

B. The Martinola Presentation

[3] The district court also found the same claims—4, 7-12, and 20—of the '411 patent to be anticipated by a public presentation made by Dr. Martinola at the International Water Conference in Pittsburgh in October 1980. Specifically, the district court found that "Martinola stated at his deposition that during his presentation at the 1980 conference he used Figure 10 of his diagram [sic] as a slide and discussed 'the use of a mixed bed ion exchange resin after hydrazine and activated carbon' for the same purposes and uses described in Ecologychem's patents." *Ecologychem*, at *40 (quoting Martinola Dep. at 18:16-19:6). Finding this to be undisputed, the district court found that this testimony established that the Martinola presentation, like his articles, anticipated the above claims of Ecologychem's '411 patent. *See id.*

[4] Ecologychem argues that "Dr. Martinola's oral presentation must stand or fall with the article as allegedly anticipatory prior art, since Dr. Martinola could not remember in 1992 what he had said twelve years earlier." Appellant's Br. at 35. We do not agree that the presentation, in and of itself, cannot anticipate claims of the '411 patent. Section 102 provides that "a person shall be entitled to a patent unless ... the invention was known or used by others in this country." 35 U.S.C. § 102(a) (1994). A presentation indicative of the state of knowledge and use in this country therefore qualifies as prior art for anticipation purposes under § 102. Furthermore, whether Dr. Martinola correctly remembered his presentation twelve years later is an issue of credibility, on which we review the district court's finding with deference. The district court found, based on

Dr. Martinola's testimony, that Figure 10 was indeed presented in 1980 by Dr. Martinola with an explanation that it was addressing the use of hydrazine. See *Ecolchem*, at *40.

The key element of the presentation, found by the district court to anticipate the claims of the '411 patent, is Figure 10. We first note that both independent claims 1 and 15 differ from independent claim 20 in contemplating that the final ion exchange step will remove not only dissolved carbon contaminants, but also any excess hydrazine that has not reacted with the dissolved oxygen in the water. Claims 4 and 7-12 (which all depend on claim 1) also require the additional step of removing the excess hydrazine. According to his deposition testimony, Dr. Martinola discussed the removal of the dissolved carbon contaminants at the 1980 presentation, but not the removal of excess, i.e., unreacted, hydrazine.⁸ This omission renders clearly erroneous the district court's finding that claims 1, 4, and 7-12 were anticipated by Dr. Martinola's presentation, and consequently we reverse that finding.

Therefore, the only claim that could be anticipated is claim 20, which claims a deoxygenation process comprising the steps of passing liquid containing oxygen and hydrazine through activated carbon, and then passing the liquid through ion exchange resins, including both strong acid cation and strong base anion exchange resins, to remove at least the dissolved contaminants. This is the exact process described by Dr. Martinola's presentation of Figure 10. We therefore affirm the district court's finding of anticipation of claim 20 of the '411 patent as not clearly erroneous.

8. Dr. Martinola testified that, during his presentation, he described his paper as showing:

[T]hat you can apply activated carbon as a catalyst and it will remove oxygen with hydrazine; but it has to be taken into account that the carbon releases salts in the demineralized water, and, afterwards, in Figure 10, there is shown a system with a catalyst column and a mixed bed.

Martinola Dep. at 18:6-12.

II. Obviousness

[5] The district court also held that the inventions of claims 1, 3-13, 15, 17, 18, and 20 of the '411 patent would have been obvious in light of the "combination of the Houghton process for deoxygenation with a mixed bed ion exchange resin to remove excess hydrazine and/or dissolved and/or undissolved carbon contaminants." *Id.* at *37. We review the district court's conclusion of obviousness *de novo*. We affirm the district court's conclusion of obviousness with regard to claim 20, and reverse the holding that obviousness was proven by clear and convincing evidence with regard to all other claims at issue.

A. The Houghton Process as a "Blueprint"

[6] The district court essentially found that the most innovative aspect of Ecolchem's process was its "[u]nearth[ing] [of] long-neglected art," holding that "Ecolchem's good fortune in obtaining the Houghton reference just as the EPRI guidelines created increased attention in the PWR industry to the problem of ambient temperature deoxygenation does not entitle Ecolchem to patent protection." *Id.* at *33.

Houghton was the co-author of a paper on deoxygenation by carbon catalysis of the reaction between hydrazine and dissolved oxygen, entitled "The Use of Active Carbon With Hydrazine in the Treatment of Boiler Feed Water" ("the Houghton article"). The Houghton article was presented at the 1957 International Water Conference at Bournemouth, England and brought to the attention of Ecolchem at the 1982 International Water Conference.⁹

9. William Miller, one of the inventors of the deoxygenation process described in the '411 patent, testified that he attended the International Water Conference in 1982 "with the intention of finding alternative deoxygenation processes, if there were any." Miller Decl. ¶ 19 (Oct. 28, 1997), J.A. at 931. By alternative, Mr. Miller was referring to processes other than deoxygenation through the use of a vacuum degasifier. While at the conference,

The Houghton process for deoxygenation of Ecolchem's process would have been obvious to one of ordinary skill in the art at the time of the invention because the Houghton process was well known in the art and the Ecolchem process was a mere modification of the Houghton process. The district court's finding that the Houghton process was not obvious to one of ordinary skill in the art is clearly erroneous. The district court's finding that the Houghton process was not obvious to one of ordinary skill in the art is clearly erroneous. The district court's finding that the Houghton process was not obvious to one of ordinary skill in the art is clearly erroneous.

The district court's finding that the Houghton process was not obvious to one of ordinary skill in the art is clearly erroneous.

Taken together, the relevant facts show that the Houghton process was well known in the art and the Ecolchem process was a mere modification of the Houghton process. The district court's finding that the Houghton process was not obvious to one of ordinary skill in the art is clearly erroneous.

Mr. Miller, manager, Jr.

Mr. Pugsley and Lightfoot for Ecolchem, one at Ft.

The Houghton article peaked the interest of Ecolochem, and its scientists began performing experiments to determine if the results predicted in Houghton's paper could be replicated in a laboratory setting. Ecolochem's scientists were skeptical, because they "had understood that hydrazine reacted very slowly with dissolved oxygen and one of [their] objectives in [the] preliminary experimentation was to run the process to determine if the Houghton process sufficiently catalyzed the reaction." Miller Decl. ¶ 22. The tests supported the Houghton article, but also revealed the presence of ionic substances in the deoxygenated water coming out of the carbon bed. Houghton did not discuss the ionic contamination. Ecolochem performed subsequent tests to assess its attempts to reduce the presence of the ionic substances, and after considerable experimentation, eventually succeeded with the patented process after more research and considerable experimentation. The district court found that "for years it had been known in the art of water treatment that activated carbon releases ionic substances into water," but that Ecolochem's scientists, who the district court found had been employed in the water treatment industry for over a decade by the time the patent issued, were unaware of this contamination at the time they conducted their experiments. *Ecolochem*, at *11.

The district court recognized that the Houghton reference did not anticipate the patent, but felt that:

Taken together, the prior art references relevant to Ecolochem's invention disclose all of the elements of the claimed invention, and their combined teachings would have suggested to one of ordinary skill in the art that the Houghton process could be followed by the use of mixed bed ion exchange resins to provide ambient temperature deoxygena-

tion and remove excess hydrazine as well as dissolved and undissolved carbon contaminants.

Id. at *34.

"A patent may not be obtained . . . if the differences between the [claimed invention] and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art." 35 U.S.C. § 103(a) (Supp. III 1997). Our analysis of the patentability of Ecolochem's invention begins with the phrase "at the time the invention was made." Here, the date of the invention is presumed to be the filing date of the parent application, December 16, 1983.

[7] In *In re Dembiczak*, we noted that: Measuring a claimed invention against the standard established by section 103 requires the oft-difficult but critical step of casting the mind back to the time of invention, to consider the thinking of one of ordinary skill in the art, guided only by the prior art references and the then-accepted wisdom in the field.

In re Dembiczak, 175 F.3d 994, 999, 50 USPQ2d 1614, 1617 (Fed.Cir.1999). We "cannot use hindsight reconstruction to pick and choose among isolated disclosures in the prior art to deprecate the claimed invention." *In re Fine*, 837 F.2d 1071, 1075, 5 USPQ2d 1596 (Fed.Cir.1988).

Our case law makes clear that the best defense against hindsight-based obviousness analysis is the rigorous application of the requirement for a showing of a teaching or motivation to combine the prior art references. See *Dembiczak*, 175 F.3d at 999, 50 USPQ2d at 1617. "Combining prior art references without evidence of such a suggestion, teaching, or motivation simply takes the inventor's disclosure as a blueprint for piecing together the prior art

some work on deoxygenation involving carbon. As a result of that conversation, Mr. Pugsley arranged for someone at Florida Power and Light to send me [the Houghton article].

Id.

Mr. Miller spoke to an Ecolochem area sales manager, John Pugsley.

Mr. Pugsley worked for the Florida Power and Light Company before he came to work for Ecolochem and he recalled that someone at Florida Power and Light was doing

to defeat patentability—the essence of hindsight.” *Id.*

[8, 9] “When a rejection depends on a combination of prior art references, there must be some teaching, suggestion, or motivation to combine the references.” *In re Rouffet*, 149 F.3d 1350, 1355, 47 USPQ2d 1453, 1456 (Fed.Cir.1998) (citing *In re Geiger*, 815 F.2d 686, 688, 2 USPQ2d 1276, 1278 (Fed.Cir.1987)). The same principle applies to invalidation. “Obviousness cannot be established by combining the teachings of the prior art to produce the claimed invention, absent some teaching or suggestion supporting the combination.” *ACS Hosp. Sys., Inc. v. Montefiore Hosp.*, 732 F.2d 1572, 1577, 221 USPQ 929, 933 (Fed. Cir.1984). Although the suggestion to combine references may flow from the nature of the problem, see *Pro-Mold & Tool Co. v. Great Lakes Plastics, Inc.*, 75 F.3d 1568, 1573, 37 USPQ2d 1626, 1630 (Fed. Cir.1996), “[d]efining the problem in terms of its solution reveals improper hindsight in the selection of the prior art relevant to obviousness,” *Monarch Knitting Mach. Corp. v. Sulzer Morat GmbH*, 139 F.3d 877, 880, 45 USPQ2d 1977, 1981 (Fed.Cir.1998). Therefore, “[w]hen determining the patentability of a claimed invention which combines two known elements, ‘the question is whether there is something in the prior art as a whole to suggest the desirability, and thus the obviousness, of making the combination.’” *In re Beattie*, 974 F.2d 1309, 1311–12, 24 USPQ2d 1040, 1042 (Fed.Cir.1992) (quoting *Lindemann*, 730 F.2d at 1462, 221 USPQ at 488).

[10] In this case, the district court used the ‘411 patent as a blueprint, with the Houghton process as the main structural diagram, and looked to other prior art for the elements present in the patent but missing from the Houghton process. The district court opinion does not discuss any specific evidence of motivation to combine, but only makes conclusory statements. “Broad conclusory statements regarding the teaching of multiple references, standing alone, are not ‘evidence.’” *Dembiczak*, 175 F.3d at 999, 50 USPQ2d

at 1617. The district court provides no support for its broad conclusory statement that it was known in the art that a carbon bed, as used in the Houghton process, would produce water with high levels of conductivity caused by the presence of ionic contaminants. Nor does the district court then provide support for its implicit finding that given water so contaminated, it would be obvious to one of ordinary skill in the art to place a mixed bed ion exchange resin downstream of the carbon bed. In fact, nowhere does the district court particularly identify any suggestion, teaching, or motivation to combine the Houghton process with a mixed bed ion exchange resin to achieve the patented process.

The district court avoids the issue, and makes implicit findings, but can point to nothing that suggests the combination of deoxygenation and demineralization processes that comprise Ecolochem’s invention. For instance, the district court finds that Ecolochem “did not try any other way to remove the ionic contaminants leached by the carbon bed other than adding the mixed bed,” *Ecolochem*, at *12, and that “[t]here is no evidence that any system for production of ultra-pure water ever included a carbon bed as the final step in water treatment.” *Id.* at *22. In addition, the district court makes the unsupported finding that “[m]any in the art knew as of 1982 that carbon beds leached contaminants which could be removed by ion exchange.” *Id.* at *21. In support of this statement, the district court relies upon the Martinola reference, and U.S. Patent No. 4,430,226 (“the ‘226 patent”) for a disclosure that “activated carbon leaches dissolved contaminants.” For the disclosure of the “removal of dissolved carbon contaminants by ion exchange”, the district court relied upon the ‘226 patent and John W. Hassler’s 1974 article “Purification with Activated Carbon,” which do state that with some types of carbon, “ion-exchange resins have been employed to remove inorganic compounds, alkalinity, or acidity not absorbable by activated carbon.” J.A. 1

1893. However, while these references teach the leaching of dissolved contaminants by activated carbon and the use of ion-exchange resins to remove carbon contaminants, neither reference suggests combining, nor provides any motivation to so combine, the two elements of the Ecolochem process, i.e., deoxygenation of the water by the Houghton process and demineralization of the water by the mixed bed.

The district court seems to find that the Martinola reference implicitly suggests the combination of the two elements, but discounts "[t]he fact that Martinola did not make Ecolochem's invention, and instead focused on the hydrogen-palladium method of deoxygenation [a]s not relevant." *Id.* at *39. The district court clearly erred in this regard. This fact is completely relevant to the obviousness analysis, since Martinola actually teaches away from combining at least one of the Martinola articles with the Houghton process to achieve Ecolochem's claimed process. While the Martinola reference describes a hydrogen and Lewatit-based deoxygenation process and mentions deoxygenation by carbon catalysis of a hydrazine/oxygen reaction, it does so only for comparative purposes. The Martinola reference actually unfavorably compares the hydrazine/carbon process, saying that it "releases salts into the demineralized water" and that the hydrogen-based process is energy saving and significantly less expensive.¹⁰

The Martinola reference is not the only reference that points to problems and concerns with the Houghton process. The Houghton article was challenged from the

day it was presented, when an audience member asked whether the process could produce water with acceptable amounts of silica. Houghton responded that contamination was a concern with his process, and both this inquiry and the response were published with the paper in a section entitled "Discussion." Another paper published contemporaneously with the Houghton article states that:

if an activated carbon-bed is used to accelerate the oxygen/hydrazine reaction, it is particularly important that no trace of carbon should enter the boiler ... [i]f it is considered essential that the hydrazine and oxygen should react before entering the boiler, then the use of ultra-violet light is a promising means of accelerating the reaction, without introducing any impurities into the system.

S.R.M. Ellis, C. Moreland, *The Reaction Between Hydrazine & Oxygen, in The Account of the Proceedings of the International Conference held at Bournemouth, 15th-17th May 1957* 8, 21 (1958). In 1960, T.F. Demmitt prepared a "Preliminary Report on the Use of Activated Carbon as a Catalyst for the Dissolved Oxygen-Aqueous Hydrazine Reaction." Demmitt stated that "[m]agnetite would be more desirable than activated carbon since there would be no tendency to 'deactivate' magnetite in filtered water," and thereby taught away from the idea of using a carbon bed as the catalyst. J.A. at 1696H. In 1962, a paper was presented at the International Water Conference, stating that research had shown two methods "to remove oxygen to sufficiently low levels with adequate capac-

zine hydrate. ... A particularly simple process, which takes place in particularly well-known stages and is also energy-saving, is the reaction between the dissolved oxygen and hydrogen gas introduced into the water. ... If we compare the final costs for [the different methods] we find that the method of oxygen reduction with hydrogen is much cheaper than the other methods. The required apparatus is also simple and needs virtually no maintenance.

Martinola at 77-81 (1980).

10. The 1980 article states:

A large number of chemical compounds can be used for reducing the elementary oxygen in water. ... Until now, however, they have merely been used for the residual degassing that follows on from thermal processes. The reasons for this are the high costs involved, the slowness to react at low temperatures and the additional introduction of salts into the water. For the elimination of oxygen in boiler feedwater and hot water, use has been made for a long time now of hydrazine in the form of hydra-

ities to be practical." Piero Sturla, *Polishing Condensate and Dearthening by Ion Exchange* at 63 (1962). Neither of these methods was based on the Houghton process. These two methods were still being used in 1977, when Culligan (a water treatment company) recommended one of the two in an internal memo for use in portable containers, and the challenges continued through the time of the invention. Even Ecolochem's scientists themselves testified that, prior to their successful tests, they did not believe they would be able to replicate the results stated in the Houghton article.

Furthermore, the district court found even the mixed bed, of which the use "to remove carbon contaminants was well known in the field," had detractors. *Ecolochem*, at *39. Edison's own engineers testified that they considered:

a four-bed [a primary cation bed, a primary anion bed, a secondary cation bed and a secondary anion bed] system superior to ... a three-bed system [a cation bed, an anion bed and a mixed bed] ... because of the problems that Edison had encountered in regenerating the mixed bed, ... and based on ... personal experience that four-bed systems generally outperformed mixed bed systems in producing pure water.

Id. at *13. There is clear evidence of teaching away in the prior art from both the demineralization process and the deoxygenation process used by Ecolochem, and no evidence that there was any suggestion in the prior art to combine these two processes, yet the district court finds the '411 patent obvious in light of the prior art.

The absence of a convincing discussion of the specific sources of the motivation to combine the prior art references, particularly in light of the strength of prior art teaching away from the use of the Houghton process, is a critical omission in the district court's obviousness analysis, which mainly discusses the ways that the multiple prior art references can be combined to read on the claimed invention. For example, the district court finds that the invention of claim 20 would have been

obvious, and that, although claims 1 and 15 differ from claim 20, "[e]ach of the additional steps of claims 1 and 15 is disclosed in the prior art." *Id.* at *22. The opinion then lists each step and states where in the cited prior art references the step can be found. This reference-by-reference, limitation-by-limitation analysis wholly fails to demonstrate how the prior art teaches or suggests the combination claimed in the '411 patent.

With hindsight, we could perhaps agree that the Houghton article seems like an obvious place to start to address the need in the power plant industry for an improved carbon-catalyzed deoxygenation process employing hydrazine that can be used commercially in a variety of applications. But, "obvious to try" is not the standard. As embodied in the '411 patent, the process would secure for the art all of the advantages that catalyzation of a reaction normally provides without prohibiting its use due to all of the incumbent disadvantages associated with the use of hydrazine. However, the incumbent disadvantages associated with the use of hydrazine to deoxygenate water as described in the Houghton article include the release of unwanted impurities into the water, including dissolved substances such as salts, organic chemicals and suspended solids. As a result, the industry widely regarded the Houghton process as too impractical to be used for large amounts of water, or for high purity deoxygenated water. Under the conditions in which PWRs operate, even minute amounts of these contaminants can adversely affect generator integrity. *See id.* at *5. Variations on this method had been tried, but discounted in favor of other deoxygenation processes. Dr. Martinola himself testified that at the time of his presentation he stated that the hydrazine method of deoxygenation led to contaminated deoxygenated water. *See supra* at I.B. Finally, the process not only worked, but worked better than expected, leading Edison to investigate "why Ecolochem's equipment can reduce [the organic concentration in the water] to less than 10

p[arts] p[er] b[illion] w[as] designed to [do so]." *Ecolochem*, at *22. Edison, in fact, gave four bidders for the HFMUD contract of Ecolochem's equipment "information about Ecolochem's process with others in violation of Ecolochem's secrecy agreement." *Id.* at *22. The sharing of information about Ecolochem's testing was a prospectively developed process used by Ecolochem and accused by Ecolochem in the '411 patent. The district court found that the bidder's ... effectively copied Ecolochem's generation process." *Id.* at *22. This evidence undermines the district court's conclusion that Ecolochem's process had been obvious to one of ordinary skill in the art.

Because we do not disavow the district court's finding on the basis for the finding of obviousness, we find that there was a suggestion, or motivation to combine the references cited against the invention, the district court's conclusion of obviousness cannot stand. The district court's finding by the district court when one of ordinary skill in the art faced with the problem of deoxygenating water for use in a nuclear power plant, as described in the Houghton article, claimed by Ecolochem in the '411 patent, would have been obvious. We have previously held that a suggestion to combine may be implicit or implicit teachings of the references themselves, from the knowledge of those skilled in the art of the nature of the problem to be solved." *WMS Gaming, Inc. v. International Game Tech.*, 184 F.3d 1385, 1397 (Fed. Cir. 1999). However, there still must be some teaching, explicit or implicit, from the skilled artisan, confronting the problems as the inventor faced, to select the elements from the prior art references for combination claimed." *In re Rouffet*, 47 USPQ2d at 1456; *see also* *Kotzab*, 217 F.3d 1365, 1366.

although claims 1 and 15 20, "[e]ach of the addi- ns 1 and 15 is disclosed *Id.* at *22. The opinion p and states where in references the step can reference-by-reference, tion analysis wholly te how the prior art ests the combination patent.

ve could perhaps agree article seems like an rt to address the need t industry for an im- alized deoxygenation hydrazine that can be n a variety of applica- is to try" is not the died in the '411 patent, ecure for the art all of catalyzation of a reac- les without prohibiting the incumbent disad- with the use of hydra- incumbent disadvan- the use of hydrazine r as described in the clude the release of into the water, includ- ces such as salts, or- suspended solids. As, widely regarded the too impractical to be- nts of water, or for- nated water. Under- high PWRs operate, s of these contami- effect generator integ- . Variations on this, ed, but discounted in- ygenation processes testified that at the on he stated that the deoxygenation led to enated water. So the process not only etter than expected- uestigate "why Eco- reduce [the organ- ater] to less than

p[arts] p[er] b[illion] with a device not designed to [do so]." *Ecolochem*, at *15. Edison, in fact, gave four prospective bidders for the HFMUD construction job a tour of Ecolochem's equipment and shared "information about Ecolochem's process with others in violation of [Ecolochem's] secrecy agreement." *Id.* Only after this sharing of information and subsequent testing was a prospective bidder able to develop the process used in the HFMUD, and accused by Ecolochem of infringing the '411 patent. The district court in fact found that the bidder's "proposed system ... effectively copied Ecolochem's deoxygenation process." *Id.* at *15. This evidence undermines the district court's conclusion that Ecolochem's process would have been obvious to one of ordinary skill in the art.

Because we do not discern any evidentiary basis for the finding by the district court that there was a suggestion, teaching, or motivation to combine the prior art references cited against the claimed invention, the district court's conclusion of obviousness cannot stand. The implicit generalized finding by the district court that, when one of ordinary skill in the art was faced with the problem of deoxygenating water for use in a nuclear power plant and the Houghton article, the combination claimed by Ecolochem in the '411 patent would have been obvious is insufficient. We have previously held that "[t]he suggestion to combine may be found in explicit or implicit teachings within the references themselves, from the ordinary knowledge of those skilled in the art, or from the nature of the problem to be solved." *WMS Gaming, Inc. v. International Game Tech.*, 184 F.3d 1339, 1355, 51 USPQ2d 1385, 1397 (Fed.Cir.1999). However, there still must be evidence that "a skilled artisan, confronted with the same problems as the inventor and with no knowledge of the claimed invention, would select the elements from the cited prior art references for combination in the manner claimed." *In re Rouffet*, 149 F.3d at 1357, 47 USPQ2d at 1456; see also *In re Werner Kotzab*, 217 F.3d 1365, 1371, 55 USPQ2d

1313, 1317 (Fed.Cir.2000) ("[A] rejection cannot be predicated on the mere identification ... of individual components of claimed limitations. Rather, particular findings must be made as to the reason the skilled artisan, with no knowledge of the claimed invention, would have selected these components for combination in the manner claimed."). Here, there was no such evidence presented. The only evidence on this issue presumes the very problem at hand—two experts testified that "if someone of ordinary skill in the art had been given the Houghton reference in 1982 and [if] they were asked to make it usable in a high-pressure power plant, they would have come up with Ecolochem's invention." *Ecolochem*, at *31 (emphasis added). The evidence available, however, indicates that if one of ordinary skill in the art had been given the Houghton reference, they would not have been inclined to use it, due to the large amount of teaching away, and the reliance in the industry on vacuum degasifiers to deoxygenate water. This finding by the district court presumes the knowledge acquired from Ecolochem's patent. We hold that the district court's finding that a skilled artisan would combine these references was clearly erroneous, and we hold that on this record the district court clearly erred in finding clear and convincing evidence of a suggestion to combine the prior art references, a suggestion to use the Houghton article as the backbone of the invention. Therefore, we reverse the district court's conclusion of obviousness with regard to claims 1, 3-13, 15, 17, and 18 of the '411 patent. As to claim 20, however, our prior decision mandates that we now undertake further analysis respecting its invalidation by the district court.

B. *Prima Facie* Case of Obviousness of Claim 20

[11] We previously held in our June 1996 non-precedential decision that: the district court clearly mischaracterized the import of Demmitt as a prior

art reference for the determination of obviousness of claim 20. Demmitt ... did not disclose the removal of carbon contaminants with an ion exchange resin. Despite the district court's mischaracterization of the importance of Demmitt, Ecolochem concedes that there is a 'prima facie case of obviousness before Demmitt and there remains one after.' However, Ecolochem argues that the secondary considerations, in this case, could rebut the prima facie case and that the district court wrongly failed to consider its evidence of secondary considerations and conclude that the evidence raised a genuine issue of fact requiring trial. We agree.... For the foregoing reasons ... the trial court's holding of obviousness of claim 20 of the '411 patent is reversed, and the case is remanded for trial on validity and infringement as to claim 20.

Ecolochem, at *4-5. Given our earlier reliance on Ecolochem's concession of the *prima facie* obviousness of claim 20, which is binding as law of the case, we must treat claim 20 separately from the other claims held obvious in the decision appealed herein. We may only conclude that Ecolochem rebutted the *prima facie* case of obviousness with regard to claim 20 if the evidence of secondary considerations supports such a holding. See *id.*; *In re Piasecki*, 745 F.2d 1468, 1471, 223 USPQ 785, 787 (Fed.Cir.1984). We therefore review the district court's findings on the evidence of secondary considerations, and, for purposes of completeness, address whether and how this evidence affects the adjudicated invalidity of all other asserted claims.

C. Evidence of Secondary Considerations With Regard to All Claims

[12] The idea that a patented invention might appear to be obvious given the excellent vision accorded by hindsight, but might not have been obvious at the time the invention was made, was discussed by the Supreme Court in *Graham v. John Deere Co.*, 383 U.S. 1, 17-18, 86 S.Ct. 684,

15 L.Ed.2d 545 (1966). In the intervening years since *Graham* a great deal of attention has been paid to the importance of secondary considerations. We discuss below many of the secondary considerations used by the courts in an effort to compensate for hindsight. The Supreme Court held that "[s]uch secondary considerations as commercial success, long felt but unsolved needs, failure of others, etc., might be utilized to give light to the circumstances surrounding the origin of the subject matter sought to be patented. As indicia of obviousness or nonobviousness, these inquiries may have relevancy." 383 U.S. at 17-18, 86 S.Ct. 684 (internal citations omitted). While we review the district court's factual findings on the secondary considerations for clear error, we review the ultimate determination of obviousness *de novo*. See *B.F. Goodrich Co. v. Aircraft Braking Sys. Corp.*, 72 F.3d 1577, 1582, 37 USPQ2d 1314, 1317-18 (Fed.Cir.1996). We find clear error in the district court's findings on several of the secondary considerations as discussed below, but upon reviewing the obviousness conclusion *de novo*, hold that even if corrected, the findings respecting secondary considerations support the district court's holding of obviousness of claim 20 in the instant case. This conclusion has no effect on the obviousness of the inventions of claims 1, 3-13, 15, 17, and 18, which we have already held would not have been obvious given the absence of any motivation to combine the teachings of the cited prior art references. Ecolochem has, however, failed to rebut the conceded *prima facie* case of obviousness with regard to claim 20. We therefore affirm the district court's conclusion that the invention of claim 20 would have been obvious.

1. Long-Felt But Unsolved Need

The district court found that "[t]he findings of fact do not support Ecolochem's contention that there was a long-felt but unsolved need to create an ambient temperature deoxygenation process for use in start up and restart of nuclear power fac-

ilities." *Ecolochem*, at *3. We hold that even if there was a long-felt but unsolved need, the "required new matter" of the claimed invention and the claim is not attenuated by the increment of demand resulting from adoption of the guidelines in 1982." *Id.*

Ecolochem argues that the guidelines merely reflected a long-felt but unsolved need for deoxygenation. It arose in the late 1970s and was only recommended, not required, deoxygenated water. Ecolochem further argues that the guidelines, other than the issuance of the guidelines for it to develop its process, it argues that the deoxygenation process was the only long-felt but unsolved need. It was not until the late 1970s that deoxygenated water during the time of the guidelines was build and install the permanent deoxygenators. Ecolochem argues that the guidelines (like emissions requirements for mobile sources) were issued only when the need was realized, and that the guidelines may have been unattainable when they were issued. Even Ecolochem pointed out that Ecolochem's process was a practical solution to the problem of deoxygenated and deoxygenated water before the completion of the

Our review of the district court's finding of no long-felt but unsolved need for clear error, a deferential standard of review. We cannot find clear error in the district court's finding that Ecolochem's process for deoxygenated water in response to the guidelines issued by EPA was not in response to a long-felt but unsolved need. The record supports the district court's finding that the guidelines urging the use of deoxygenated water were issued in 1982, and that Ecolochem filed for a patent on its process on December 16, 1982. The evidence supports the district court's finding that Ecolochem's process was not in response to a long-felt but unsolved need in the power industry, but in response to a long-felt but unsolved requirement imposed by the guidelines.

ities." *Ecolochem*, at *35. It went on to hold that even if there had been such a need, the "required nexus between the claimed invention and the long-felt need is attenuated by the increased market demand resulting from adoption of the EPRI guidelines in 1982." *Id.*

Ecolochem argues that the 1982 EPRI guidelines merely reflected the acknowledged need for deoxygenated water that arose in the late 1970s and that the regulations only recommended, rather than required, deoxygenated water. Ecolochem further argues that there were reasons other than the issuance of these regulations for it to develop its processes. Furthermore, it argues that its mobile chemical process was the only way to meet this long-felt but unsolved need for deoxygenated water during the time required to build and install the permanent deoxygenators. Ecolochem argues that the guidelines (like emissions requirements for automobiles) were issued only after a long-felt need was realized, and that the regulations may have been unattainable at the time they were issued. Even Edison in its brief pointed out that Ecolochem had the only practical solution to the need of SONGS for deoxygenated and demineralized water before the completion of the HFMUD.

Our review of the district court's finding of no long-felt but unsolved need is for clear error, a deferential standard of appellate review. We cannot say the district court's finding that Ecolochem produced demineralized water in response to the guidelines issued by EPRI to be clearly erroneous. The record shows that the guidelines urging the use of deoxygenated water were issued in 1982, and Ecolochem filed for a patent on its deoxygenization process on December 16, 1983. This evidence supports the district court's finding that Ecolochem's process was developed not in response to a long-felt need in the power industry, but in response to a short-term requirement imposed by EPRI's guidelines.

2. Commercial Success

[13] We have previously held that a party cannot "demonstrate commercial success, for purposes of countering the challenge of obviousness, unless it can show that the commercial success of the product results from the claimed invention." *J.T. Eaton & Co. v. Atlantic Paste & Glue Co.*, 106 F.3d 1563, 1571, 41 USPQ2d 1641, 1647 (Fed.Cir.1997). We have further held that a presumption arises that the patented invention is commercially successful "[w]hen a patentee can demonstrate commercial success, usually shown by significant sales in a relevant market, and that the successful product is the invention disclosed and claimed in the patent." *Id.* In the instant case, Ecolochem offered evidence that its invention was practiced at 28 plants and generated almost \$13 million in revenue from 1983 to 1990. Once Ecolochem made the requisite showing of nexus between commercial success and the patented invention, the burden shifted to Edison to prove that the commercial success was instead due to other factors extraneous to the patented invention. *See id.*

At trial, Edison countered that the commercial success of Ecolochem's process was due solely to the fact that it was part of a mobile apparatus, and that since none of the claims at issue include such a mobility limitation, the commercial success factor favors a finding of obviousness in the instant case. The district court found that both Edison and Ecolochem had carried their respective burdens, finding that the volume of Ecolochem's sales satisfied the requirements to show commercial success, but also that Edison proved the commercial success was due to factors other than those claimed. The district court explained:

Ecolochem's limited commercial success with its patented process was primarily attributable to (1) Ecolochem's ability to meet the need in the PWR industry for short-term emergency services through its Mobile Flow service; (2) Ecolochem's

ability to provide deoxygenation services while PWR plants built permanent deaeration equipment to meet new EPRI standards; (3) Ecolochem's head start in marketing mobile deoxygenation treatment services through use of its patented Mobile Flow trailer; (4) the increased blowdown rates that resulted from the 1982 tightening of EPRI guidelines for salts in the steam generator. In short, Ecolochem's commercial success was due not to the nature of the claimed invention, but to other economic and commercial factors unrelated to the technical quality of the patented process.

Ecolochem, at *36.

The district court's finding, however, ignores Edison's own statements to the contrary. Edison indicated in its briefs that it chose Ecolochem because the "first vendors Edison hired provided poor quality water and constantly shuttled demineralization trucks on and off Edison's property to meet Edison's capacity requirements." Appellee's Br. at 3. Edison's brief also states that Ecolochem's process "enabled it, unlike Edison's previous vendors, to regenerate its trailers on-site and provide purer quality water at the large volumes Edison needed." *Id.*

These statements make it clear that the commercial success of Ecolochem's product was, in fact, based on two factors: the improved filtration process, and the mobility of the commercial embodiment. Edison did not differentiate the improved filtration process from the patented process in any way, focusing only on the missing limitation of mobility in the claims at issue. The success was due to both the mobility, undisputedly not covered by the claims, and to the improved filtration process, undisputedly covered by the claims. Edison had the burden of disproving that the improved filtration process contributed to the success of the invention, and its own brief undermines its argument, by conceding the benefits of Ecolochem's filtration process. See *Ryko Mfg. Co. v. Nu-Star, Inc.*, 950 F.2d 714, 716, 21 USPQ2d 1053, 1055 (Fed. Cir.1991) ("To overturn a patent, the challenger must clearly prove those facts

which support patent invalidity."). Consequently, the district court clearly erred in finding that Edison met its burden of proving that the success of the invention was due to factors not claimed in the patented invention.

3. Failure of Others to Make Invention

In evaluating this factor, the district court held that an unsolved problem is not evidence of non-obviousness unless skilled workers in the art have tried and failed to solve the problem. See *Ecolochem*, at *36. The district court then held that Ecolochem did not show that others had tried but failed to make Ecolochem's invention. See *id.*

Ecolochem cited a 1986 internal business memorandum circulated at Arrowhead, a competitor of Ecolochem's. In this memorandum, under the heading "*OPPORTUNITY*," Arrowhead wrote the following: "Ecolochem claims a proprietary process for deoxygenation and has exploited this market with little or no interference from Arrowhead. Markets exist primarily at the nuclear plants. Several approaches for deoxygenation exist..." J.A. at 3049.

[14] Edison, in turn, responded with testimony from its employees that when Edison discussed its deoxygenation needs with contractors in 1982, only two contractors mentioned chemical deoxygenation: Arrowhead and Ecolochem. Edison employees further testified that vacuum degasifiers were the industry standard, were reliable and economical, and that every other contractor stated that it intended to deoxygenate with a vacuum degasifier. There was no testimony as to whether Arrowhead succeeded in making a working chemical deoxygenation device. The district court evaluated Edison's testimony, and the absence of any testimony contradicting it, and found that no competitor had attempted to employ a chemical deoxygenation process. The failure of others is therefore reduced to a credibility question: whether the district court believed the Edison employees. "This court gives gr

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deference to the district court regarding credibility. *roll Touch, Inc. v. Sys., Inc.*, 15 F.3d 157, 1836, 1842 (Fed.Cir.1991). *City of Bessemer*, 575-76, 105 S.Ct. 151 (1985)). We cannot find evidence on the part of the district court that it found no evidence of Edison's failure to make a chemical deoxygenation process.

4. Simultaneous Invention

[15] "The fact of new invention, though not determinative of obviousness, is somewhat constitutive of the level of the art." *The Int'l G. States*, 187 Ct.Cl. 376, (1969). "[T]he possibility of simultaneous invention by two talented inventors working independently... may or may not be obviousness when considered in the circumstances." *Lin. v. Wood*, 1460, 221 USPQ at 487.

The district court found that the issuance of Ecolochem's patent for the Hook of NWT independent Houghton process with its exchange resin, thereby invalidating Ecolochem's invention. *Ecolochem v. Hook*, at *36. Ecolochem's Hook testified at trial that the idea embodied in his invention, which combined the Houghton process and ion exchange resin, but Dr. Sawochka's. Dr. Sawochka, an acknowledged expert in the field, testified that the system sketch and test apparatus embodying the Houghton process is irrelevant to obviousness or nonobviousness. *Id.* at 103. Competing inventor Dr. Sawochka may show bias.

Appellant's Br. at 56. Edison testified that "as both Sawochka and Hook testified, Hook was left largely responsible for performing his project." *Id.* at 53.

deference to the district court's decisions regarding credibility of witnesses." *Carroll Touch, Inc. v. Electro Mechanical Sys., Inc.*, 15 F.3d 1573, 1580, 27 USPQ2d 1836, 1842 (Fed.Cir.1993) (citing *Anderson v. City of Bessemer City*, 470 U.S. 564, 575-76, 105 S.Ct. 1504, 84 L.Ed.2d 518 (1985)). We cannot find it clearly erroneous on the part of the district court to have found no evidence of failure by others to make a chemical deoxygenation device or develop such a process.

4. Simultaneous Invention

[15] "The fact of near-simultaneous invention, though not determinative of statutory obviousness, is strong evidence of what constitutes the level of ordinary skill in the art." *The Int'l Glass Co. v. United States*, 187 Ct.Cl. 376, 408 F.2d 395, 405 (1969). "[T]he possibility of near simultaneous invention by two or more equally talented inventors working independently, ... may or may not be an indication of obviousness when considered in light of all the circumstances." *Lindemann*, 730 F.2d at 1460, 221 USPQ at 487.

The district court found that "prior to the issuance of Ecolochem's patent, Todd Hook of NWT independently combined the Houghton process with a mixed bed ion exchange resin, thereby independently developing Ecolochem's invention." *Ecolochem*, at *36. Ecolochem argues that Mr. Hook testified at trial that the:

idea embodied in his test apparatus, which combined the hydrazine/carbon process and ion exchange, was not his but Dr. Sawochka's. Dr. Sawochka, an acknowledged expert in the field, provided the system sketch and configured the test apparatus embodying the idea. Obviousness or nonobviousness to experts is irrelevant to obviousness under Section 103. Competing innovation by Dr. Sawochka may show bias but nothing more.

Appellant's Br. at 56. Edison responds by stating that "as both Sawochka and Hook testified, Hook was left largely on his own in performing his project." Appellee's Br. at 53.

The issue of simultaneous invention is directly tied to the level of knowledge attributable to one of ordinary skill in the art. The district court weighed the testimony discussed above, and determined that there was evidence of simultaneous invention, and the fact that the one who performed the experiment was supervised by one of extraordinary skill in the art did not enhance the former's level of knowledge. Essentially, the district court found that this secondary consideration factor favors obviousness. As this was based on the district court's determination that Dr. Sawochka's and Mr. Hook's testimony was credible, we must give that finding great deference. Consequently, we cannot discern clear error in the finding of simultaneous invention by the district court.

5. Teaching Away

[16] The district court found that "Ecolochem presented no evidence that the prior art expressed skepticism concerning the efficacy of using the Houghton process or of combining the Houghton process with ion exchange. Likewise, the Court's findings of fact establish that the prior art did not teach away from Ecolochem's invention." *Ecolochem*, at *37. Ecolochem argued that the Houghton process had been around for decades and was not well known or regarded, citing a number of articles stating concerns with the Houghton process.

We previously found, in our unpublished June 1996 decision, that Ecolochem had put forth evidence "that various references taught away from the invention as they warned against the Houghton process because of the carbon contaminants." *Ecolochem, Inc. v. Southern Cal. Edison Co.*, 1996 WL 297601, at *5. We discussed above the fact that Martinola teaches away from the Houghton process, as that reference found the process inefficient and expensive. We also discussed above the history of prior art teaching away from Houghton. Based on the body of evidence provided to the district court, we find

clearly erroneous the district court's determination that the prior art did not teach away from using the Houghton process or from combining it with a mixed bed in any manner.

6. Copying and Acclamation

[17] Another indicia of non-obviousness of a product is the acclamations it receives when it is released, and the copying that occurs. The district court found "that [although] Ecolochem's process was warmly received in the water treatment industry and was copied by at least two competitors, that copying did not result from the novelty of Ecolochem's invention and is of little weight in assessing obviousness." *Ecolochem*, at *37.

[18] Ecolochem argues persuasively that its device was copied because it was significantly less expensive than vacuum degasifiers, the only alternative in the field. Furthermore, testimony from Edison indicates that Ecolochem's process was better than its competitors'. The district court gives little credit to Ecolochem's arguments, finding only that the process was not novel. It is a factual determination as to what the exact reason for the copying was, but here we hold that it was clear error on the district court's part to discount the copying because the court believed that the process was not novel. Therefore, this factor cuts against a conclusion of obviousness.

[19] We note, however, that a showing of copying is only equivocal evidence of non-obviousness in the absence of more compelling objective indicia of other secondary considerations. See *In re GPAC*, 57 F.3d 1573, 1580, 35 USPQ2d 1116, 1122 (Fed.Cir.1995) ("[M]ore than the mere fact of copying by an accused infringer is needed to make that action significant to a determination of the obviousness issue.") (quoting *Cable Elec. Prods. v. Genmark, Inc.*, 770 F.2d 1015, 1028, 226 USPQ 881, 889 (Fed.Cir.1985)). The reason is that the alleged copying "could have occurred out of a general lack of concern for patent property." See *Cable Elec. Prods.*, 770

F.2d at 1028, 226 USPQ at 889. Consequently, even though the district court clearly erred in stating that the copying was not connected to the patented aspects of the invention, its error does not carry great weight in our evaluation of the obviousness of the invention of claim 20 in light of all the secondary considerations, combined with the other evidence and findings on the prior art.

7. Weighing of the Factors

We must now weigh all the secondary considerations to determine whether the district court's error in not finding commercial success and teaching away, when aggravated by its error in failing to find copying; renders erroneous its conclusion that the invention of the claims at issue here would have been obvious. The district court did not clearly err in finding that there was no long-felt but unsolved need, that there was no failure by others to make the invention, and that there was a successful simultaneous invention by others. As explained above, the district court clearly erred in finding that the commercial success of Ecolochem's product was based only upon its mobility; that the prior art did not teach away from the Houghton process; and that there was no evidence of copying. However, only two of these factors are actually stated in *Graham*; the other falls under *Graham*'s "etc." clause. The factors specifically mentioned in *Graham*, and those that we give the most weight to in the instant case, are the commercial success of the invention, long-felt but unsolved needs, and failure of others to invent.

In our *de novo* obviousness review, we hold that the absence of any evidence that others were trying to emulate the patented process, and the fact that within two years of the publication of the EPRI guidelines Ecolochem had applied for a patent on the process, is more indicative of obviousness than the evidence of modest commercial success, and the teaching away from the use of the unmodified Houghton process is indicative of non-obviousness. The question of obviousness in the instant case is

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close one, but we hold that the considerations, taken as a whole, overcome the other evidence.

Our earlier unpublished decision placed the burden on Ecolochem to show that on all the secondary considerations, taken as a whole, the case is a *prima facie* case of obviousness as to claim 20. We cannot say that the district court erred in concluding that Ecolochem has not rebutted its case of obviousness with claim 20.

However, for the remainder of the issue, claims 1, 3-13, 15, the secondary consideration of commercial success is one of several factors, all of which are assessed in determining obviousness. See *Graham*, 383 U.S. at 17-18. Given the absence of any other evidence, we hold that the remaining claims were not proven to be clear and convincing evidence of obviousness over the prior art.

close one, but we hold that the secondary considerations, taken as a whole, do not overcome the other evidence of obviousness.

Our earlier unpublished decision placed the burden on Ecolochem to rebut, based on all the secondary considerations, the *prima facie* case of obviousness with regard to claim 20. We cannot say that the district court erred in concluding that Ecolochem has not rebutted the *prima facie* case of obviousness with regard to claim 20.

However, for the remaining claims at issue, claims 1, 3-13, 15, 17, and 18, the secondary consideration evidence is but one of several factors, all of which must be assessed in determining obviousness. See *Graham*, 383 U.S. at 17-18, 86 S.Ct. 684. Given the absence of any proof of a motivation to combine, we hold that the remaining claims were not proven obvious by clear and convincing evidence in light of the prior art.

CONCLUSION

For the reasons stated above, we affirm the district court's holding that it was proven by clear and convincing evidence that the invention of claim 20 would have been obvious and was anticipated, but reverse the district court's holdings that claims 1, 3-13, 15, 17, and 18 were proven invalid either as anticipated and/or obvious. We remand this case for award of damages, consistent with the district court's finding of willful infringement which stands. The decision, therefore, is

AFFIRMED-IN-PART, REVERSED-IN-PART, and REMANDED.

COSTS

Defendant shall pay costs.

